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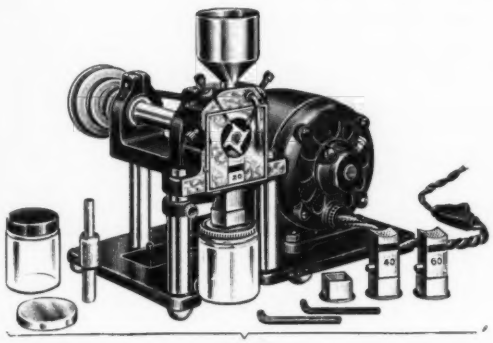

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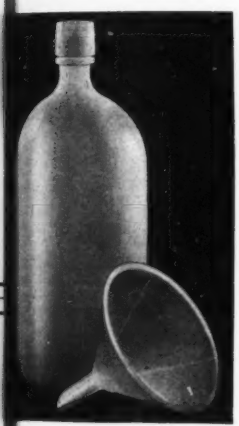

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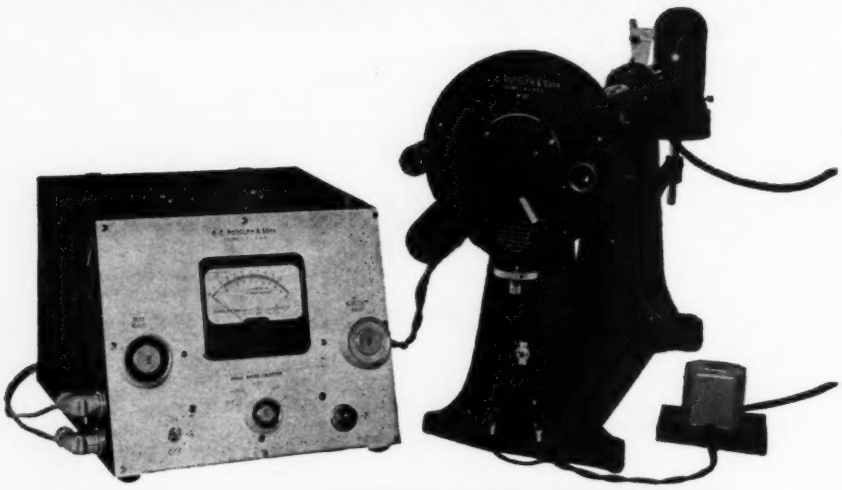

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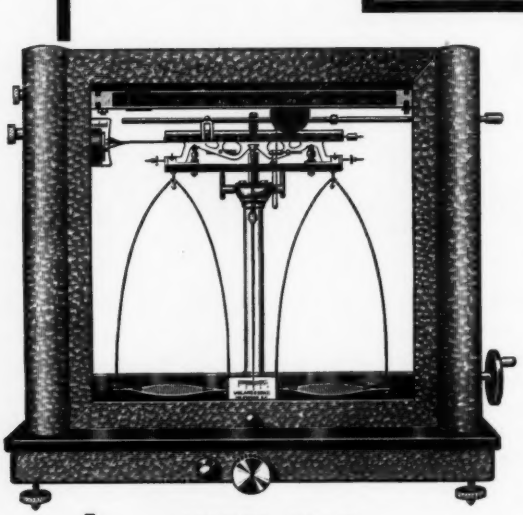

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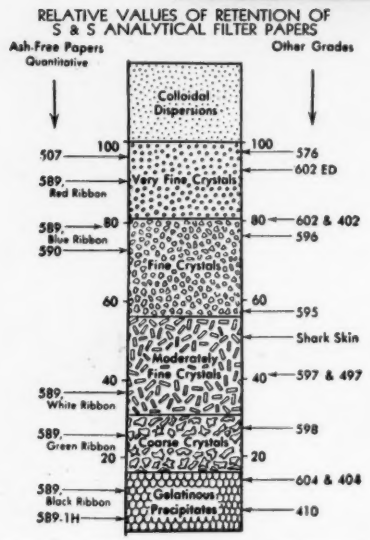

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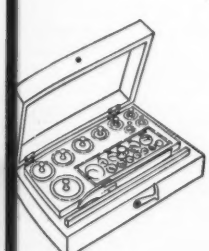

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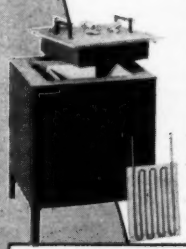

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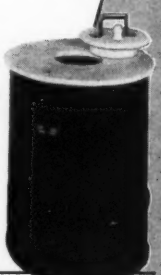

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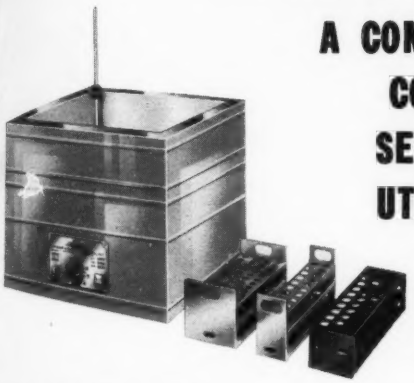

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CE-3002			8½" L x 14" W x 13" H	1 Wasserman 2 Kahn 3 Army	115 V 50-60 Cy. 500 Watts	Room to 100°C	95.00
CE-3005	Serological —or— Utility	13" L x 12" W x 5" D with 3½" Diffuser Plate —or— 13" L x 12" W x 7" D with 1½" Diffuser Plate	15" L x 14" W x 13" H	3 Wasserman 4 Kahn 6 Army	115 V 50-60 Cy. 1000 Watts	Room to 100°C	125.00
CE-3007			15" L x 14" W x 13" H	3 Wasserman 4 Kahn 6 Army	115 V 50-60 Cy. 1000 Watts	Room to 100°C	125.00
CE-3010	Serological —or— Utility	26" L x 12" W x 5" D with 3½" Diffuser Plate —or— 26" L x 12" W x 7" D with 1½" Diffuser Plate	28" L x 14" W x 13" H	6 Wasserman 8 Kahn 12 Army	115 V 50-60 Cy. 1500 Watts	Room to 100°C	175.00
CE-3012			28" L x 14" W x 13" H	6 Wasserman 8 Kahn 12 Army	115 V 50-60 Cy. 1500 Watts	Room to 100°C	175.00

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CE-3002 P	6¼" x 11¾" x 1½"	To give utility depth of 7"	3002	10.00
CE-3005 P	12¾" x 11¾" x 3½"	To give serological depth of 5"	3005	15.00
CE-3007 P	12¾" x 11¾" x 1½"	To give utility depth of 7"	3007	15.00
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iso-Amyl Acetate
n-Amyl Chloride
Aniline
Azoxybenzene
Benzenesulfonyl Chloride
Benzylamine
Benzyl Mercaptan
p-Bromophenacyl Bromide
o-Bromotoluene
m-Bromotoluene
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sec-Butyl Alcohol
n-Butyl Bromide (Pract.)
n-Butyl Chloride
n-Butyl Ether
p-tert.-Butylpyrogallol
iso-Butyraldehyde
p-Chlorobenzenesulfonic Acid
β-Chloropropionic Acid
o-Chlorotoluene
Cinnamyl Alcohol
β-Dextrose Pentaacetate
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3,5-Dinitrobenzoyl Chloride
2,4-Dinitrophenol
Diphenylbenzidine
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p,p'-Tetramethyldiaminodiphenylmethane
β-Mercaptoethanol
o-Methoxybenzoic Acid
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Methylene Bromide
Methylene Chlorobromide
Methyl Chloroform
6-Methylnicotinic Acid
Methyl Orange
Methyl Red
o-Nitroaniline
m-Nitrobenzaldehyde
p-Nitrobenzaldehyde
p-Nitrophenol
n-Octyl Acetate
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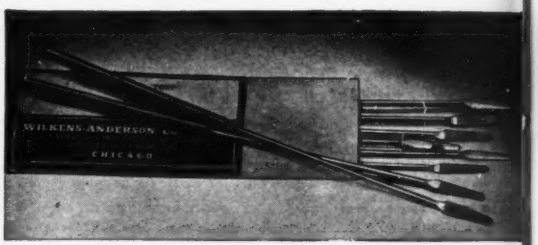

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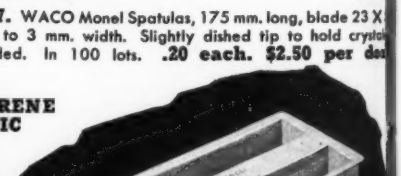

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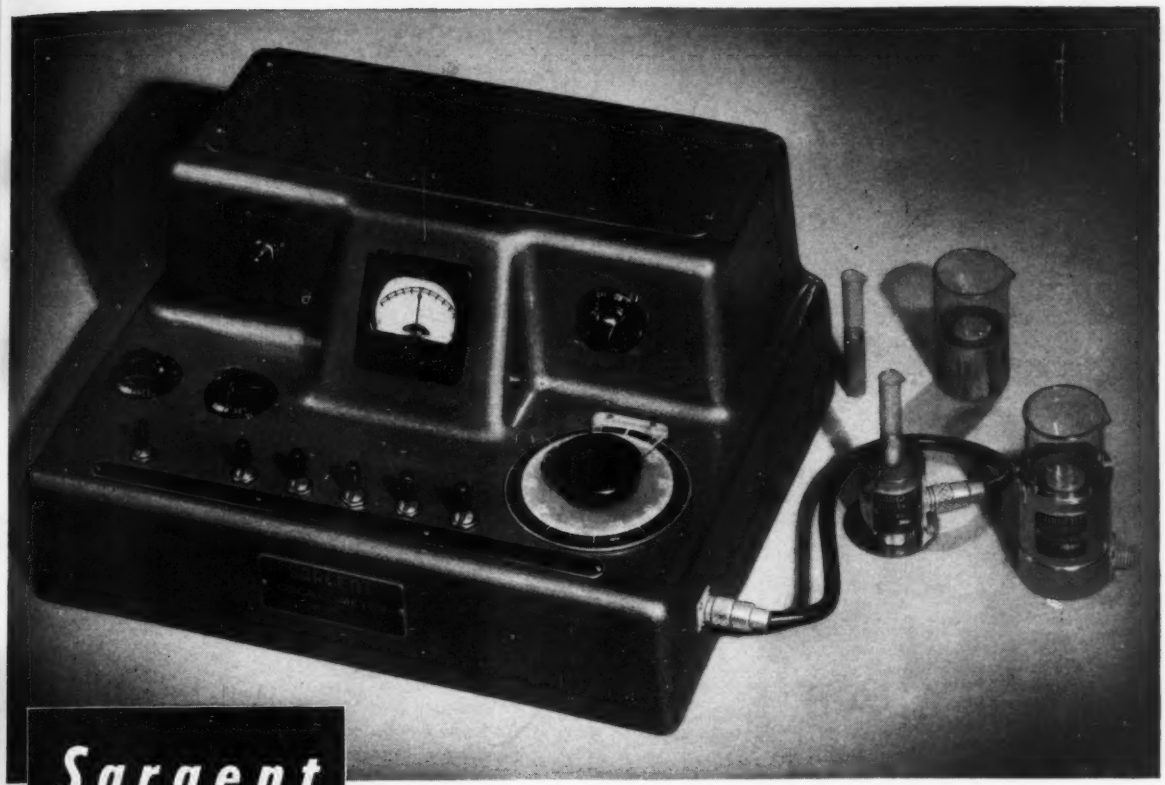

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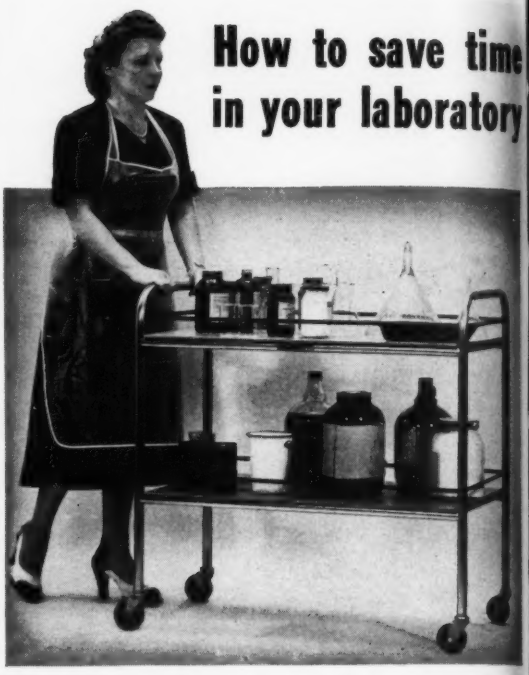

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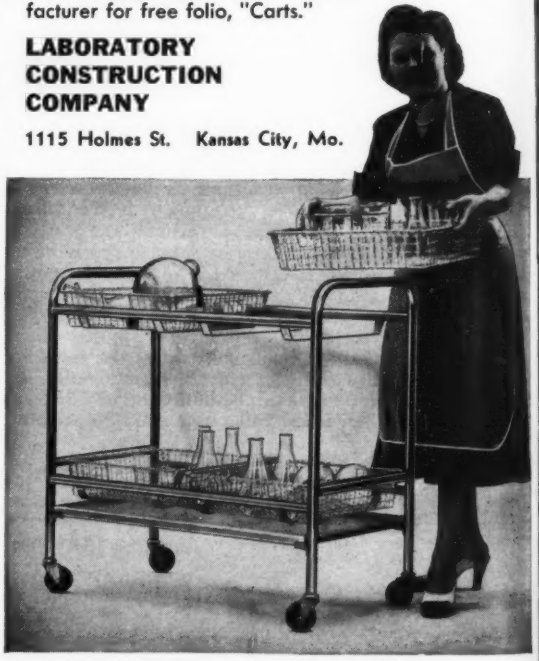

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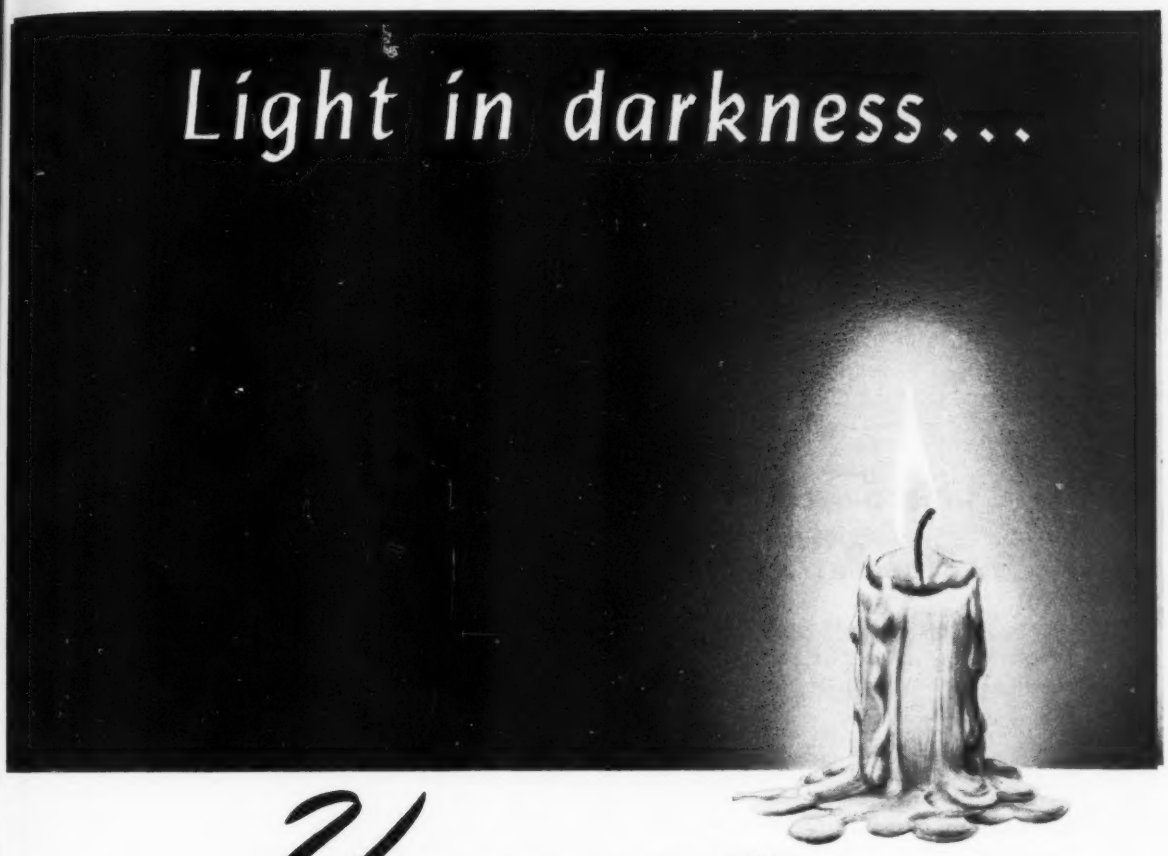

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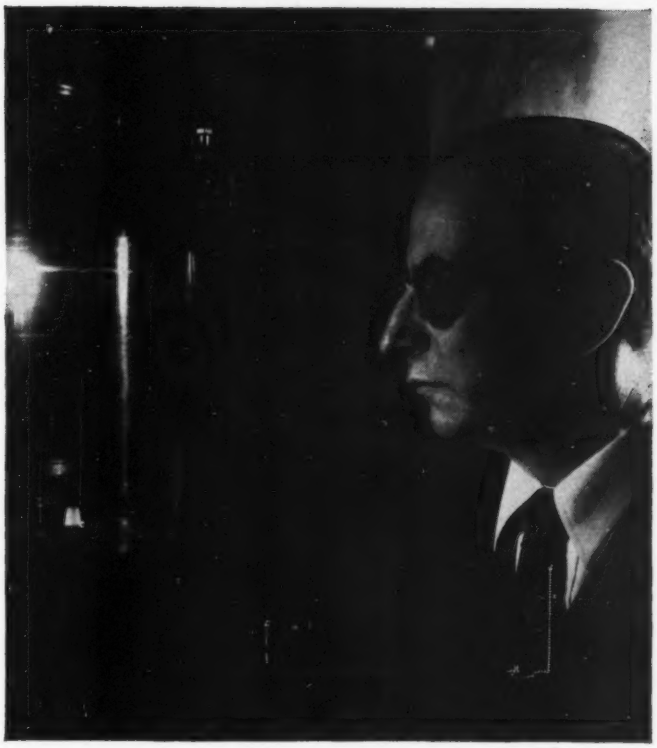
INVENTOR-SCIENTIST



One of the world's foremost authorities on the behavior and control of the electric arc. He left his job as mathematics instructor at Cornell University in 1916 to work as a coil winder in the Westinghouse East Pittsburgh, Pa., plant. But his brilliant handling of engineering problems won immediate attention. In 1922 he was named head of the general research section, four years later Research Consulting Engineer, and in 1938 was appointed Associate Director of the Research Laboratories.

His colleagues at the Westinghouse Research Laboratories say of Dr. Joseph Slepian that "he can look at an electric arc and see not fire and heat, but all of the atoms, ions, and molecules arranged in a neat mathematical formula". They also say that if you want to know anything about arcs, Slepian is your man.

Dr. Slepian's work with the electric arc hasn't remained in the realm of pure mathematics, however, for he combines with it a practical knack for invention that has produced some 225 patentable ideas thus far in his career. This prolific record has prompted one of his associates to remark that "if Dr. Slepian takes a pencil out during lunch, it's almost a sure bet another patent is in the making".

He developed the "De-ion®" circuit breaker and the "De-ion" protector tube, which have helped pave the way for transmission of power at higher voltages and for the greatly improved defense of power lines against lightning. To cite just one instance, before "De-ion" flashover protectors were installed on a 47-mile stretch of line in a western state, there were 46 interruptions a year because of lightning. Afterwards, interruptions averaged less than two a year.

Similarly, Dr. Slepian's study of arc behavior led to the development of the Ignitron mercury-arc rectifier. Perfected in the 1930's, the Ignitron came into its own in 1940 when the requirements of aluminum production reached an all-time high. Now Ignitron installations provide the direct-current power for magnesium and aluminum plants the nation over. The Ignitron has also been adapted as the control element in electrical circuits that generate power for two of the nation's largest cyclotrons. And its most recent application is in the field of electrified locomotives, where it promises greater simplicity and economy of operation.

A keen and agile thinker, Dr. Slepian likes nothing better than to joust with younger researchers on scientific topics. One of his favorite hobbies is to devise plausible but impossible inventions and then challenge his colleagues to find the flaw.

Of the many honors bestowed on Dr. Slepian, nearly all have stressed the happy combination of pure science and practical inventiveness. It is the kind of combination that at Westinghouse has made for the continuous flow of new and improved equipment, while providing a fruitful source for the products of tomorrow. Westinghouse Electric Corporation, Pittsburgh, Pennsylvania. G-10226

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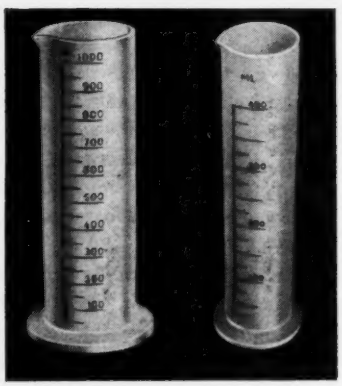

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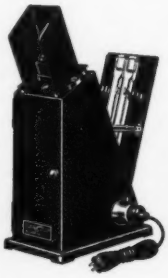

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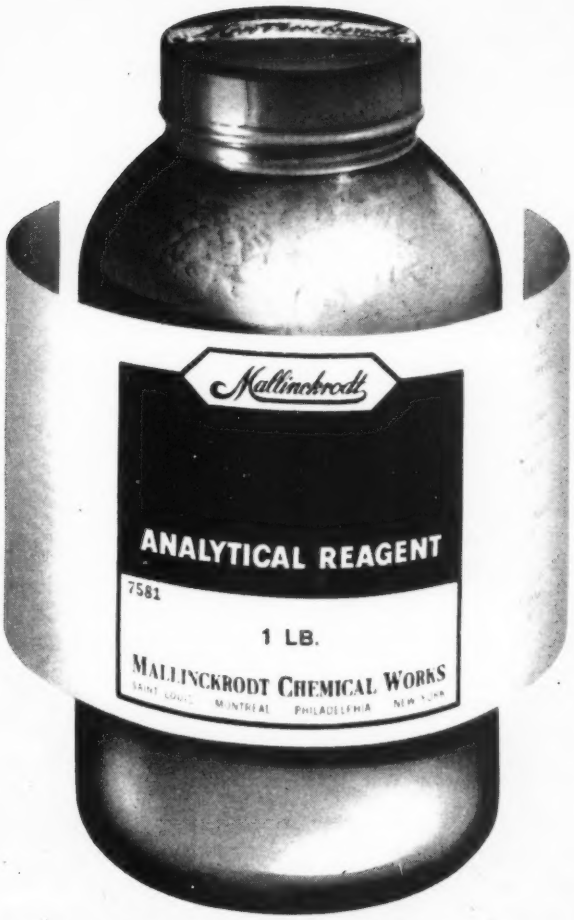

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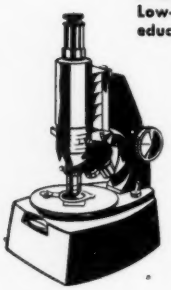

(American Optical.) Has reverse position for clear visibility, built-in polaroid analyzer, compensator slot, single focusing adjustment, rotating stage and built-in illumination. Complete with double nosepiece and cabinet. Wide selection of objectives, eyepieces and compensators available at extra charge.

18561T.....$158.00

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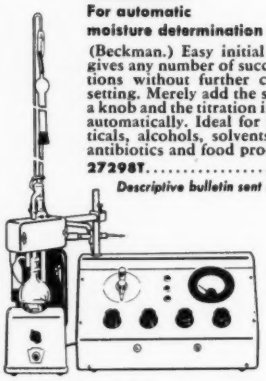
For automatic moisture determination



(Beckman.) Easy initial adjustment gives any number of successive titrations without further checking or setting. Merely add the sample, turn a knob and the titration is completed automatically. Ideal for pharmaceuticals, alcohols, solvents, tobaccos, antibiotics and food products.

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Length	5½"	14"	13
Immersion depth	3"	8"	7
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8721T

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28046T

No.	Centipoise Range	Each
1	20-85	$16.50
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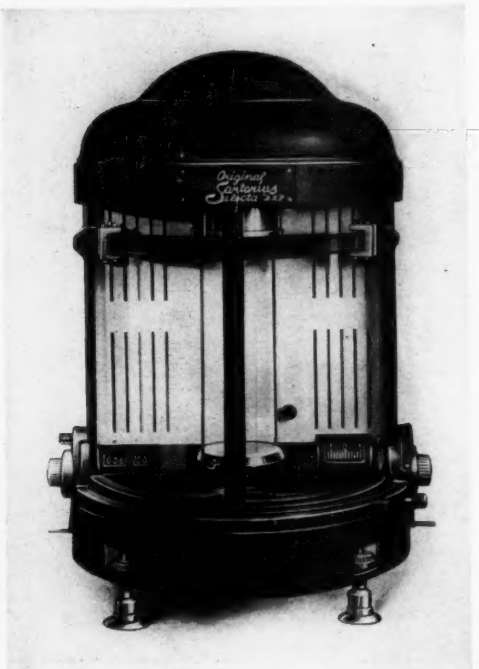

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by Mary Elvira Weeks

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"Included in the book are more than 350 illustrations, many of which show original apparatus. Much of this in its crudity serves the more to emphasize the enormous achievement produced with it."

"On the whole it can be said that this book should be of interest to both layman and chemists. It will serve as a reference or equally as a source of information to those curious about investigations made about the building matter from which everything material surrounds them."

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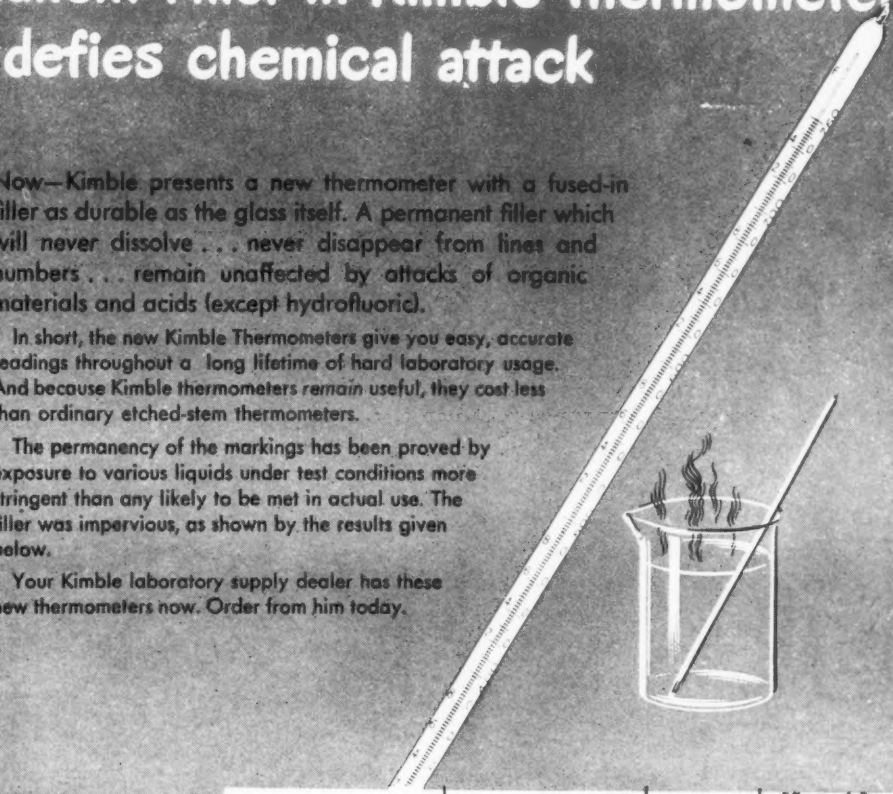

Liquids	Temperature of Exposure	Hours of Exposure	Effects of Exposure on Filler	
			Liquid	Vapor
Glacial Acetic Acid	100°C.	1265	None	None
20.24% Hydrochloric Acid	100°C.	1265	"	"
Nitric Acid Concentrated	100°C.	1265	"	"
Sulphuric Acid "	100°C.	1265	"	"
Cleaning Solution (H_2SO_4-Dichromate)	100°C.	1265	"	"
Aqua Regia	Room Temp. (25°-30°C.)	1265	"	"
Fuming Sulphuric Acid	Room Temp. (25°-30°C.)	1265	"	"
Toluene	Boiling (111°C.)	306	"	"
Varsol	Boiling (165°C.)	306	"	"
Turpentine	Boiling (150°C.)	306	"	"
Carbon Tetrachloride	Boiling (77°C.)	306	"	"
Methyl Ethyl Ketone	Boiling (80°C.)	306	"	"
Iso-amyl Acetate	Boiling (142°C.)	306	"	"
Aniline	150°C.	306	"	"

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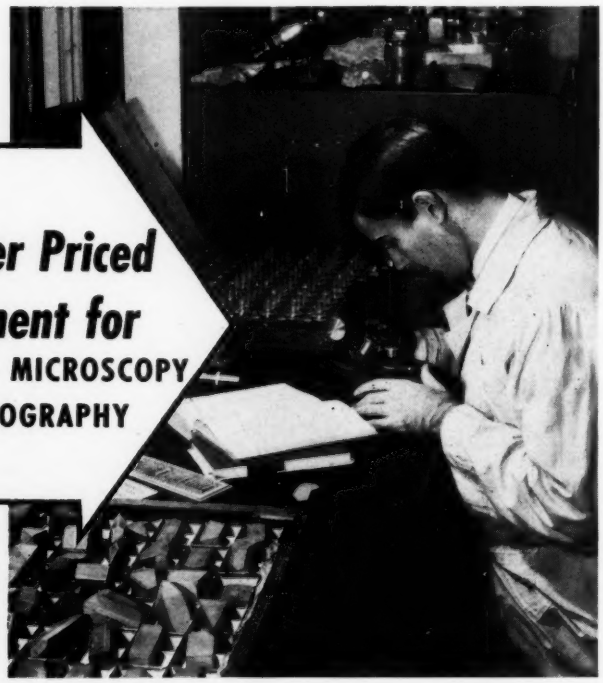

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"Any chemist at all interested in studying Russian should be much encouraged on reading Dr. Perry's chapter entitled Suggestions for Study Methods. Here the approach to study of Russian is analyzed logically and clearly. And he will certainly want to take advantage of all the useful material packed so solidly into the thoughtfully organized chapters on Vocabulary Problems, Inorganic Chemical Nomenclature, Organic Chemical Nomenclature, and Russian Grammar, as well as the Glossary of Technical Terms which contains almost 2,000 words. In fact, this book would be a good investment even to those chemists who want to learn only enough Russian to read the titles of technical articles."

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HEATING ELEMENT—Heavy Nichrome V resistance wire, spread over entire base of inside chamber. Positive temperature uniformity assured.

DOOR—Fully insulated, hung on a continuous hinge and fitted with a fool-proof latch designed to insure safety. Exclusive dovetail design gives positive seal.

PILOT LIGHTS—Red neon light denotes unit operation, green neon light for thermostat operation.

MODEL SN-4440—A most popular oven, 12" x 12" x 12" inside. Temperatures to 232 deg. C. (100 to 450 deg. F. range). Two adjustable Stainless Steel shelves, 6-foot cord and plug. For 110 volt, 60 cycle, single phase A.C. current. 800 watts..... **$117.50**

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MODEL SN-4500—Similar to SN-4480. Stainless Steel interior but baked enamel exterior, and aluminum coated shelves. For 110 volt, 60 cycle, single phase A. C. current. 1750 watts..... **$175.00**

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Out of the Editor's Basket

Adjustable Stem Thermometer

Scientific Glass Apparatus Co., Inc., Bloomfield, New Jersey, has just announced the development of a new adjustable stem thermometer called "Adjustatherm" which is made in two different stem lengths—$6^1/_2$ and $11^1/_2$ inches—for flasks ranging from 250 to 22,000 ml. Both sizes are available in three ranges: 0° to 150°C., 0° to 250°C., and 0° to 360° C.

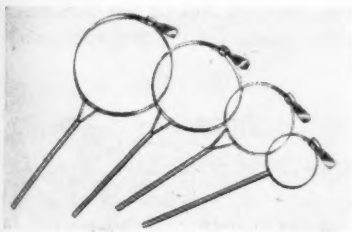
It is claimed that a set of two "Adjustatherms" will replace up to nine conventional type thermometers. It is further claimed that in addition to taking the liquid temperature, you may also obtain the vapor temperature without dismantling the equipment. You simply lift up the thermometer.

The "Adjustatherm," which works like a trombone, consists of a thermometer having either an etched scale with clearly defined markings against a yellow background, or a milk-glass scale. The thermometer fits into a precision bore sleeve (or adapter) with either a standard taper or semiball joint.

For complete information, including prices, write to the Scientific Glass Apparatus Co., Inc., Bloomfield, New Jersey.

New Varigrip Clamps

Made of corrosion-resistant stainless steel, the new Labline Varigrip clamp holds beakers, Dewar flasks, vacuum vessels etc. It may be used as a funnel holder or support ring to support extraction apparatus, heating mantles, gas cylinders, distillation columns, resin reaction kettles, and large glass tubing.



Varigrip is always rigid and is quickly and easily adjusted with a large flat thumb screw. The combination handle and clamp removes hazards when pouring or handling beakers containing hot or dangerous liquids.

Varigrip clamps are available in four sizes. Additional details may be had from Labline, Inc, 219 North Desplaines Street, Chicago 6, Illinois.

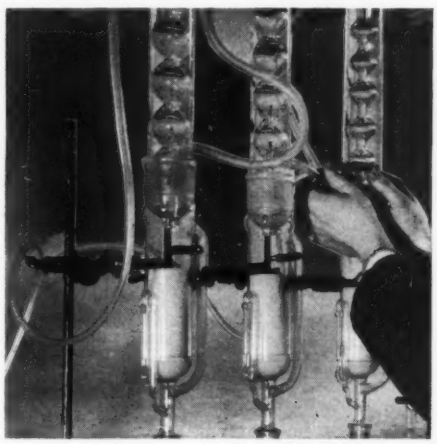
Cork-Boring Board

Christian S. Rondestvedt, Jr., of the University of Michigan, suggests the use of rubber tiles for the protection of cork borers and bench tops. Boards about 2 × 3 ft., are made from ordinary flooring-grade rubber tiles cemented to felt, which in turn is cemented to a plywood base, or directly to the bench top. The rubber is almost impervious to cork borers.

Micro-Microammeter

Beckman Instruments, Inc., South Pasadena 5, California, has announced the development of a highly stable micro-microammeter (Beckman Model V) which measures extremely small currents with great accuracy and convenience. It will measure direct currents from 0.3 microampere to 0.3 micro-microampere full scale, with an absolute accuracy better than 5 per cent in all ranges.

The exceptional performance of this instrument results from a special vibrating reed condenser used as the capacity modulator. Zero drift is typically less than 0.0005 volt in a 24-hour period, the reproducibility of 1 per cent is unaffected by ambient temperature or line voltage variations, and currents as low as 3×10^{-15} ampere can be reliably detected.



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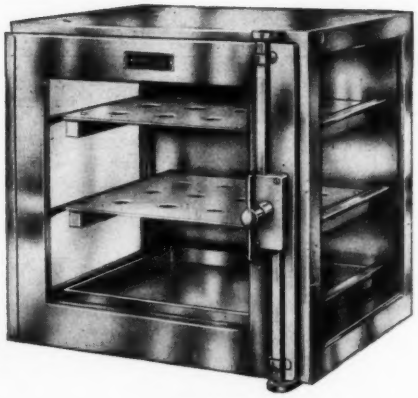

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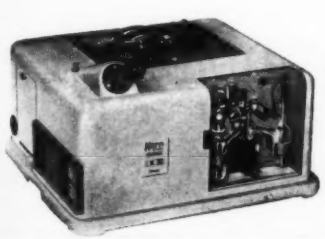

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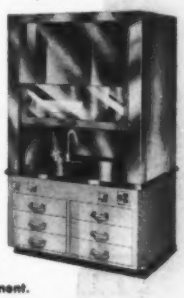

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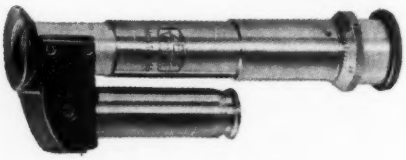

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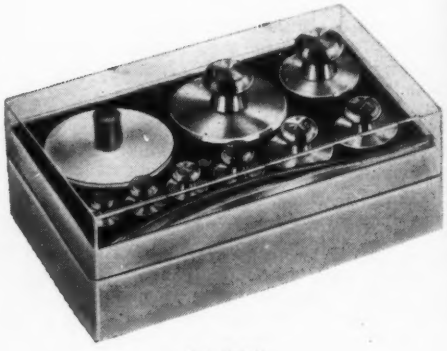

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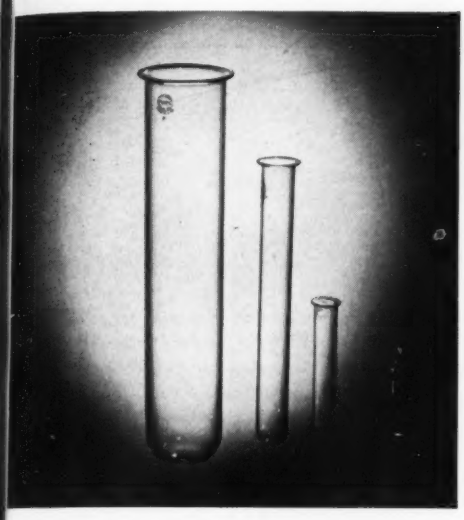

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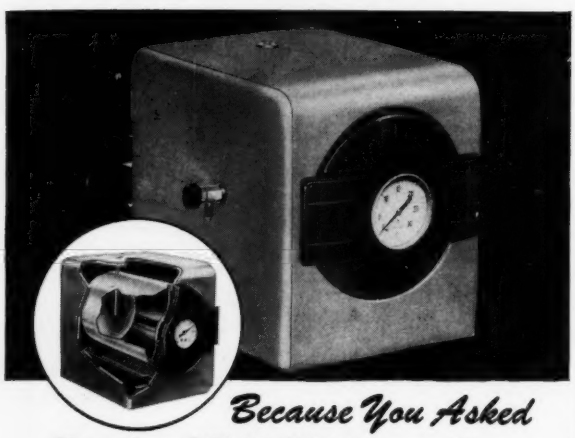

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*Gilmont, R., *Anal Chem.* **20** 474, (1948)

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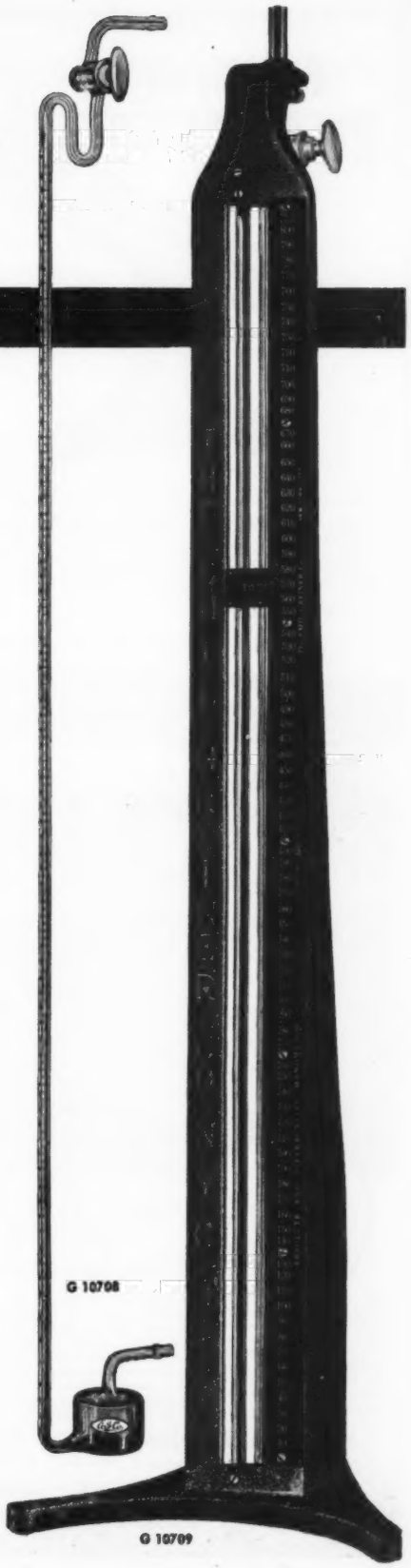

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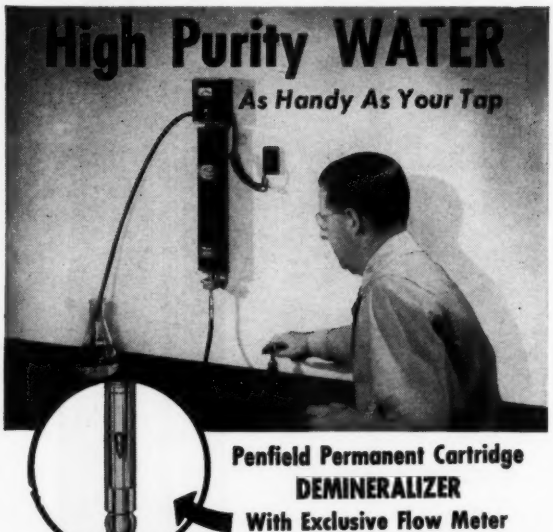

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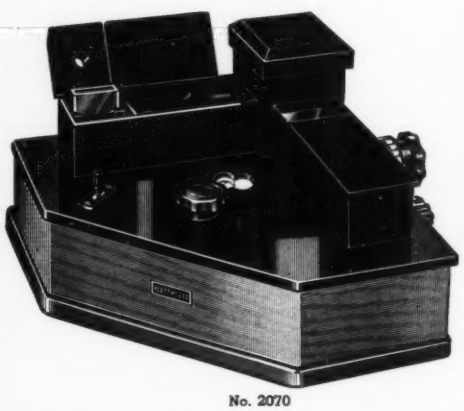

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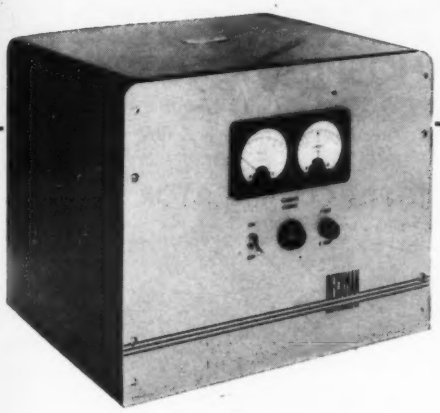

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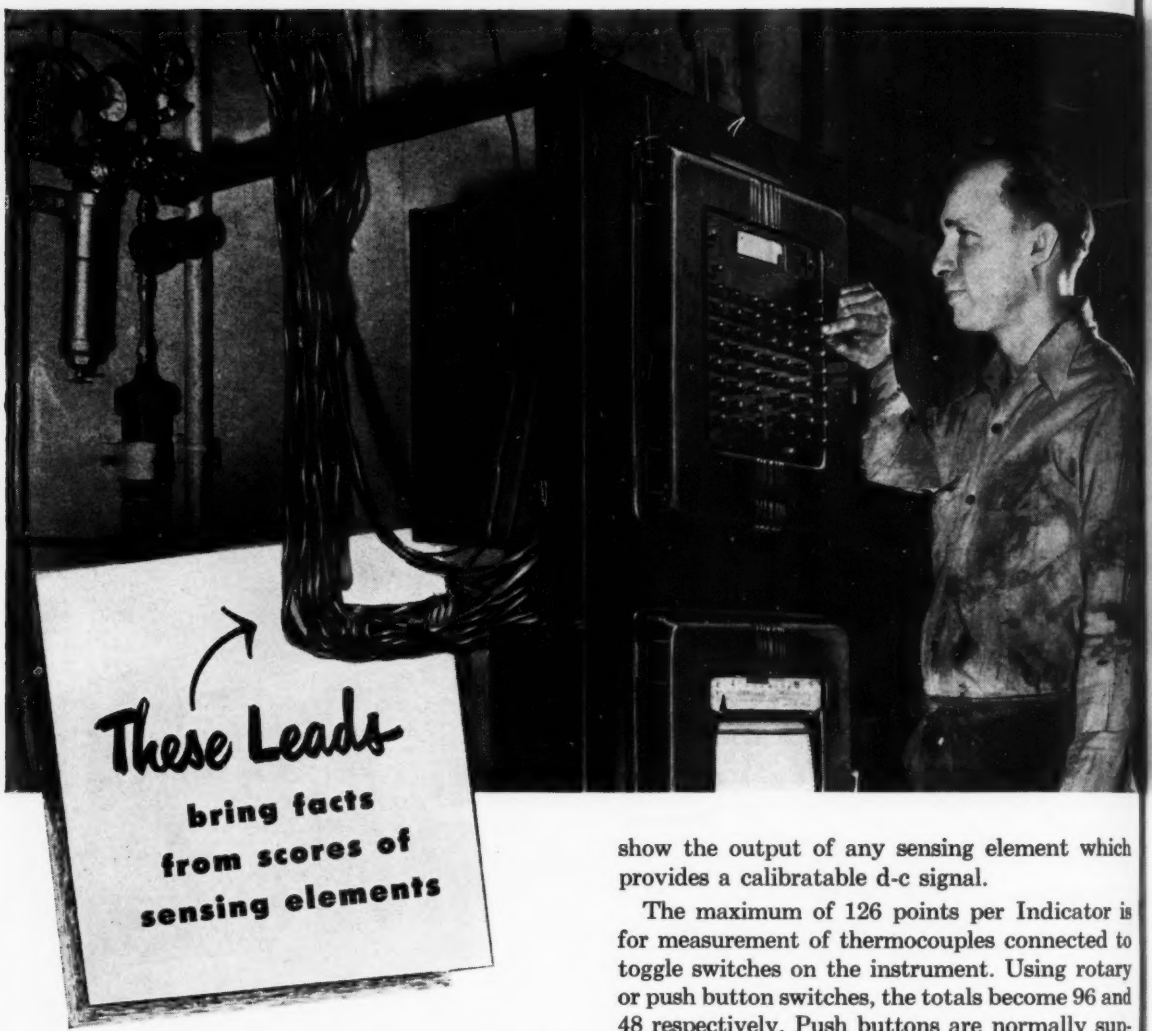

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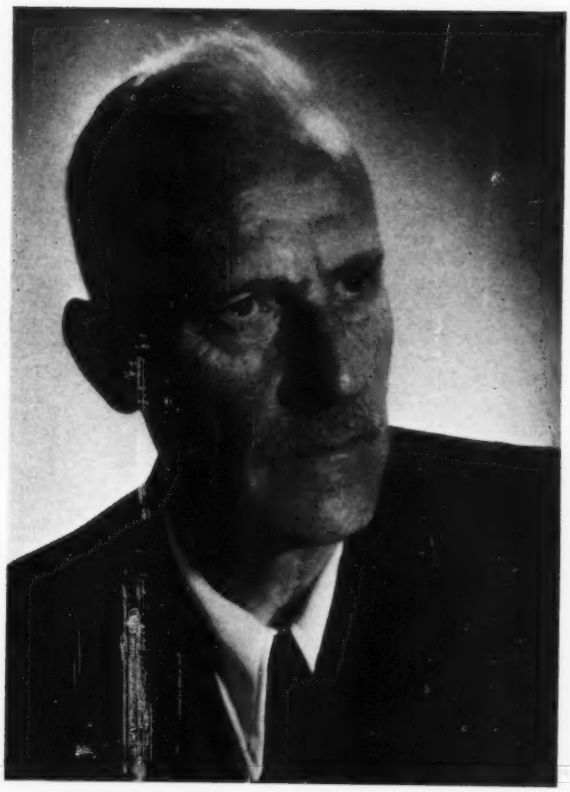

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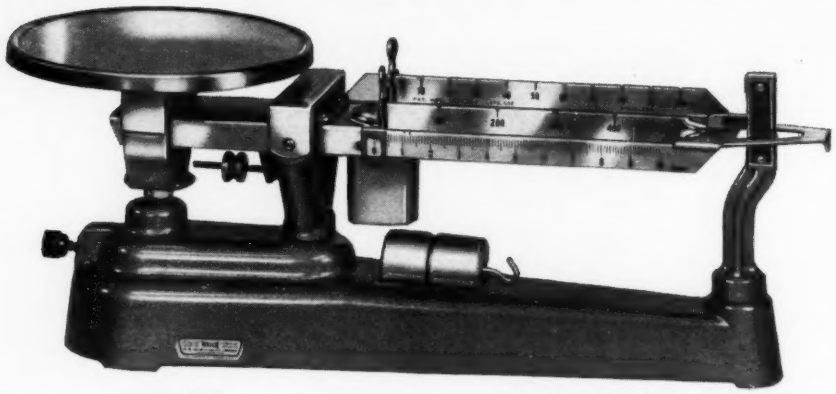

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Editor's Outlook

MANY of our readers will very likely skip over the article on page 443 of this issue (on the Harcourt Butler Technological Institute) without noticing that an object lesson is hidden in it.

Although India was a major contributor to the science and technology of chemistry in the early days of their history, and although much good research in pure science is now going on in that country, nevertheless, in comparison with most of the western world, her state of technological development is unfortunate.

Still, it does not pay to adopt an attitude of lofty superiority. Civilization in India was at a high level when our own forbears, in the forests of Europe, were very likely brandishing clubs and wearing animal skins. Such an unwarranted attitude is indeed one of the reasons why Americans are now hated in so many quarters.

According to tradition, the Indian mind prefers to deal with the intangible—even the impractical. However, in the present case it has come down to earth and is grappling with intensely practical, local problems. One gets the impression that this is rather a hard psychological limitation for the Indian mind.

The bright young men in our own schools will glance at the illustrations and perhaps smile at the primitive facilities shown. They certainly do suffer by first comparison with the elaborate equipment usually pictured in our technical schools.

But the bright young men overlook two facts: First, that in every field technology had to *begin* with primitive equipment, and some of the most important work ever done, in both pure and applied science, was carried out with the facilities of a kitchen or a simple workshop. No doubt, the "recapitulation" theorem has its application in scientific as in biological evolution.

Second, and more vital, is the likelihood that many young men, when they take their first jobs in industry, will be faced with a similar necessity to improvise with the simplest materials. Not every industrial chemical plant has a well-equipped laboratory (which may, indeed, be unfortunate) and ambitious young chemists may often find their best opportunities for advancement in the small, struggling concerns where they have little chemical competition. Here they may find it necessary to start technological research with an enameled pail, three feet of garden hose, and (if lucky) a buret. It will be hard going if their training has "conditioned" them to the automatic titrimeter, the spectrophotometer, and the magnetically damped analytical balance.

This is no criticism of the many fine modern instruments. Students should be familiar with their use and applications, but one of their great advantages is the opportunity they offer of learning how to get along without them! I remember the sage remark of an old teacher of mine: "By the time a piece of equipment gets into the hands of the apparatus makers all the interesting work with it has been done." A little exaggerated, perhaps, but it contains a germ of truth. Beware lest we become a profession of button-pushers!

IONOGRAPHY: A NEW FRONTIER IN ELECTROPHORESIS

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Stritch School of Medicine, Loyola University
Chicago, Illinois

In the past, two general methods have been available for studying the electrophoretic behavior of charged particles in a liquid. The microscopic method, which depends on the direct observation of the motion of the particles with a microscope, is adapted to the study of mobilities of fairly large particles. Its usefulness was extended somewhat by finding that various finely subdivided inert materials such as glass, oil, collodion, etc., can in some instances be so completely covered with adsorbed protein that they behave as if they are large protein particles.

In the moving-boundary method the displacement of the particles in an electric field is recorded photographically as the movement of a boundary between a dispersion and the dispersion medium. The method has found particular usefulness in the study of protein systems. The heart of the apparatus is essentially a U-tube in which buffer solution can be layered carefully over the protein-buffer dispersion. Sharp boundaries can be maintained if thermal convection currents are minimized by careful temperature control and if the protein-buffer has been previously dialyzed against the buffer itself so that the electrolyte concentration does not change appreciably across the boundary. An adequate description of the microscopic and moving boundary techniques has been given by Briggs (*1*).

Recent work has shown that a third method of achieving electrophoretic separations and of determining electrophoretic mobilities is also workable. This newer technique depends on the electromigration of charged particles, either ions or colloidally dispersed substances, on wet surfaces of filter paper or similar materials. The extent of movement is determined by such procedures as developing colored derivatives, incorporating radioactive tracers, using light-absorptive characteristics, or determining changes in the electrical properties of the paper ribbon. The method, for which the name ionography has been suggested (*2*), appears to be more flexible than the microscopic or moving-boundary techniques. It lends itself equally well to the study of electromigration of ions and colloidal materials, and has already scored considerable success in the fractionation, separation, and identification of such complex systems as mixtures of amino acids and proteins.

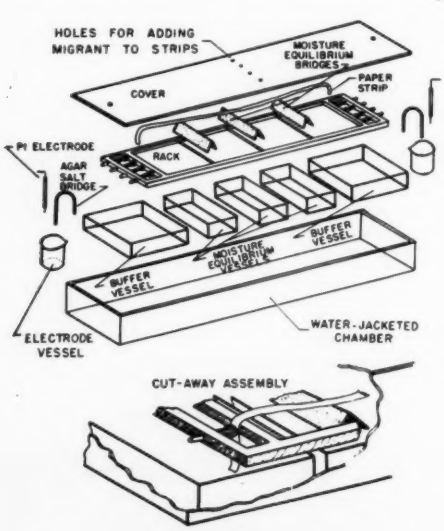

Figure 1.
A Multiple-strip Apparatus for Ionography

EXPERIMENTAL METHODS

Although several simple early forms of the ionograph were satisfactory for qualitative work, it was soon realized that chromatographic interference had to be eliminated if the quantitative potentialities of ionography were to be realized. A schematic diagram of an apparatus which goes far toward achieving this objective is shown in Figure 1. It consists of five principal component parts: (1) A removable frame for holding the paper strips, (2) Two buffer vessels into which the ends of the paper strips dip, (3) Two electrode vessels, containing the electrodes and connected by salt bridges to the buffer vessels, (4) Three moisture equilibrium bridges or "feeders," (5) An enclosing water-jacketed chamber.

The Bakelite frame (20 × 85 cm.) can accommodate several strips of filter paper, 90 cm. in length, measured from the surface of the liquid in one buffer vessel to the surface of the liquid in the other. A small spirit level was used to adjust it to a horizontal position. The strips are held in a taut, horizontal position by plastic draw-bolts, actuated by stainless steel coil springs, at either end of the frame. In later models of the apparatus the frame is adjustable so it may be used to hold large square sheets of paper, for two-dimensional experiments, as well as paper ribbon of various lengths. The buffer vessels consist of glass trays (approximately 2.5 × 20 × 25 cm.), each holding about one liter of buffer solution. The removable frame rests on the edges of the buffer vessels and the ends of the paper strips dip into the buffer solution.

The electrode vessels are 200-ml. beakers containing the same buffer solution as in the buffer vessels, and connected to them by agar-KCl salt bridges, through openings in the cover of the water-jacketed chamber. Platinum wire electrodes are used. The salt bridges normally contain 3 per cent sterilized agar in 1.5 *M* KCl solution.

The three moisture equilibrium bridges or "feeders" consist of rectangular sheets of filter paper (8 × 16 cm.) which are folded sharply along the median line. The folded paper sheets are opened to an angle of about 30° and inverted over thin glass rods so that the edges of the paper dip into narrow glass troughs (moisture equilibrium vessels) filled with buffer solution. The ends of the glass rods are held in place in the side bars of the removable Bakelite frame at intermediate points, as shown in Figure 1. The moisture equilibrium bridges and the auxiliary buffer troughs are not connected into the electrical circuit. Their function is to help maintain a uniform water sheath on the paper strips. In some experiments, especially those conducted at low temperature and of shorter duration, or at low ionic strengths, good results may be obtained without the feeders in place.

The vapor-tight Lucite chamber incloses (*1*) the frame which holds the paper strips, (*2*) the buffer vessels, and (*3*) the moisture equilibrium bridges and auxiliary buffer troughs. The chamber is double-walled throughout so that water may be circulated through it to maintain constant temperature in the interior.

The apparatus is prepared for use in the following manner. The paper strips (unless otherwise stated, Eaton and Dikeman, No. 613, 8 mm. in width) are placed in the Bakelite frame. The paper strips are then wetted by allowing 2 ml. of buffer solution to trickle onto each strip, care being taken to distribute the buffer as evenly as possible over the whole length of the filter paper. After pulling the paper strips taut, the moisture equilibrium bridges are wetted with buffer solution and enough of the solution is poured into each of the auxiliary buffer troughs to bring the liquid level up to that of the solution in the buffer vessels. The final leveling is done by means of a manifold syphon. The prepared frame is now placed in the chamber so that the ends of the filter paper dip into the buffer vessels.

In order to avoid chromatographic and diffusion effects, it is essential that the tautness of the filter paper strips be adjusted so that they are essentially level and rest lightly on the moisture equilibrium bridges. It is also necessary to wet the paper strips to the proper degree. The most consistent and reproducible results are obtained when the paper strips are wetted to the point where they have a slight sheen.

When the frame is in place, the chamber is closed and the salt bridges, electrode vessels, and electrodes are set into position. The air in the chamber is replaced with helium or hydrogen, saturated with water vapor at a temperature slightly higher than that of the buffer solutions. The hydrogen and helium, being excellent conductors of heat, accelerate the dissipation of the heat arising in the paper strips due to the electrical current (*3*). Evaporation from the paper strips is thus reduced to a minimum, and, in turn, chromatographic interference is largely eliminated. The hydrogen or helium also aids in maintaining the temperature of the paper strips close to that of the buffer solution and the circulating water in the jacketed chamber. Because of the explosion hazard involved in handling hydrogen, helium is used in most experiments. In general, when the electrical energy developed, per paper strip, exceeds 0.25 watt at 25°C., the use of helium is recommended. As the temperature is reduced from 25° to zero, the need for helium decreases rapidly, so that when experiments are run in the neighborhood of 0°C. good results are obtained using a simple air atmosphere.

When the ionic strength of the buffer solution saturating the paper strip exceeds 0.1, however, it becomes difficult to achieve linear movement of the migrant with time. Under such adverse conditions, runs have been made over a period of 4 to 28 hours. The curve obtained when the raw mobilities, y, are plotted against time, t, can be represented very closely by an empirical equation. When the curve is extrapolated, analytically, back to zero time it is found to yield the same mobility for the migrant as that determined by the moving-boundary method, under identical conditions of buffer, ionic strength, pH, and temperature (*28*).

Another technique is sometimes used to minimize evaporation, when for some reason a buffer ionic strength approaching 0.1 must be employed, especially in the neighborhood of room temperature. Glycerol, or some similar low molecular weight non-electrolyte, is added to the buffer saturating the paper strips, to comprise 5–15 per cent of the solution volume. A decided decrease in evaporation of water from the strip is observed, but the sharpness of the zone boundaries on the ionogram is lowered and some minor complications in dyeing and drying the paper strips are introduced.

It should be mentioned that measurements on the moving-boundary apparatus are usually made at buffer ionic strengths exceeding 0.1, while ionographic measurements to be used for mobility studies are generally best made at buffer ionic strengths of considerably less than 0.1,—a value of 0.05 or less being a common figure.

After equilibrium conditions have been established inside the chamber, with the current on (about 15 to 30 minutes), the stoppers in the cover are removed singly, and a small amount of the solution containing the migrant is added to each paper strip at previously marked points. It is best applied with a calibrated micropipet, as a thin streak (0.5 mm. or less) across the full width of the paper ribbon. For amino acids and proteins, from 0.005 to 0.01 ml. of a solution of the migrant (3 to 8 mg. per ml.) is usually sufficient.

As an alternative procedure, in many experiments, especially in the neighborhood of 0°C. the hinged cover

is opened, and the migrant is added at several previously marked points on each strip, except one. This latter strip is marked at the corresponding points with bromophenol blue. Since the mobility characteristics of the latter substance are now well established, its movement is used to detect any erratic behavior throughout the length of the strips, *i. e.*, it serves as a control.

The potential is supplied by a variable-voltage vacuum tube rectifier, operating from the 110-volt a.-c. line. In earlier experiments, 90-volt radio B-batteries served as the source of potential. A sensitive milliammeter is placed in the circuit in series with the paper strips.

The veronal buffer solutions used in these experiments were prepared in the manner described by Michaelis (*4*). The other buffer systems were made up as described by Clark (*5*). The pH of the buffer solutions in the buffer vessels was checked before and after each run, to make certain it did not change during the course of the experiment. Also the pH along the length of the paper strips was determined in selected instances, several times during the course of the experiment. To do this, narrow-range pH-indicating paper was used as well as a Beckman Model G pH meter equipped with a surface type (No. 1190–42) glass electrode. After a run, the paper strips were dried in a current of warm air.

To determine the displacement of amino acids the dried paper strips are sprayed with a solution of 0.1 per cent ninhydrin in butanol. To detect the displacement of proteins the paper is dried on a glass plate by directing a stream of hot air over it, or, in some cases, by placing it in an oven, set at 105°C., for several minutes. It is then passed, for about 0.5–1.0 minutes, through a solution prepared by saturating 95 per cent ethyl alcohol with mercuric chloride and dissolving one gram of bromophenol blue in each 100 ml. of the solution. The strip is then dried as before.

A series of three or four 100-ml. beakers containing a saturated aqueous solution of mercuric chloride are now set out in a row. The paper is dipped repeatedly, for about a 30-second interval at a time, in each beaker in succession, until the saturated mercuric chloride solution in the last beaker remains water-clear on dipping the strip into it. The paper ribbon is now dried. The zone on the ionogram which represents the area covered by the protein now appears as a dull greenish-blue area. If the paper strip is then passed back and forth over an open beaker containing concentrated ammonium hydroxide, the zone changes to a sharp, deep-blue color. In general, the displacement of the leading edge of the band on the ionogram, from the forward edge of the initial zone of spreading, is measured. From this measurement, the mobility of the migrant (in microns/second per volt/cm.) is computed.

Quantitative Experiments. In earlier paragraphs, two important practical factors in achieving linearity of movement of the migrant with time were mentioned. They were, first, the employment of agar-filled salt bridges to separate the products of electrode reactions from the buffer vessels into which the ends of the paper strips dipped, and second, the use of helium to help dissipate the heat developed in the paper strips. In addition to these, a number of other conditions must be met if this technique is to be utilized for quantitative experiments. To be suitable for mobility studies the region of the filter paper through which the migrant moves must be uniform with respect to water sheath, ionic composition, potential gradient, and temperature. Uniformity in these factors is reflected in the constancy of the reading on the milliammeter in series with the paper strips, and by linear movement of the migrant with respect to time. In the case of proteins, it was generally found advisable to dialyze the samples against the appropriate buffer, before making a run.

It is of prime importance that the paper strips be held horizontally throughout their whole length and that the level of the liquid in all the vessels within the chamber be identical. When the paper strips were allowed to sag, non-linearity of movement with time was observed, probably due to chromatographic and other effects. Durrum (*6*) has reported that he was not able to obtain linearity of movement with time, in similar experiments, when the center of the paper strip was elevated above the level of the ends of the paper. The reason for establishing a uniform and constant liquid level in all the inside vessels is to prevent capillary syphoning from one vessel to another through the paper strips, with its concomitant chromatographic interference. In some experiments of long duration, a manifold syphon with a bore of small diameter was allowed to remain in place during the course of the experiment. The syphon apparatus thus insured the maintenance of a constant liquid level in all the vessels within the chamber, despite the fact that a differential existed in the amount of water transported by positive and negative ions through the paper strip.

The criterion of "linearity of movement with time" is used to test out the apparatus whenever experiments of a more quantitative character are contemplated. Without observing the conditions noted above, only qualitative results are usually obtainable. It was found possible to achieve linear movement with time using a variety of amino acids and proteins (*7*), and with colored compounds such as bromophenol blue and N-(3,5-dinitrophenyl)-glycine, or dyed proteins. The colored substances were found especially suitable for this purpose since they could be followed visually from one end of the paper strip to the other.

MOBILITY

Despite the superficial resemblance of ionography to paper chromatography, a careful distinction should be maintained between the two separation processes. In chromatography, separation of solution constituents depends on a distribution between a mobile and a less-mobile or nonmobile phase. In ionography, separation results from the migration of ions or charged

particles at different rates, in an electrical field, rather than from distribution equilibria, adsorption-desorption effects, or counter-current exchanges. The paper strip serves as a carrier for the solvent or dispersion medium and helps to fix the migrating substances in position when the current is turned off, until the paper is dried. Every effort is made to reduce chromatographic interference to a minimum.

Historical. Following the advent of Tiselius' new apparatus, numerous publications brought out the importance of electrophoresis as a research tool. There were, however, certain needs, such as absolute separations and the handling of small ions, which were not very practicable on the moving-boundary apparatus. It was these needs which gave impetus to the development of electromigration through electrolytes stabilized with such substances as ground glass wool (*8*), asbestos fiber (*9*), silica gel (*10*), and agar (*11*). In these techniques, the preparation of the stabilized column is usually quite time-consuming, the removal of separated fractions of such materials as protein hydrolysates is not easy and a very large element of chromatography is present. This latter factor is large because the migrant is forced to move through the stabilizing agent as well as through the electrolyte. Various combinations of chromatography and electrophoresis have also been developed (*12, 13*), but the technique of ionography as described here is of relatively recent origin.

Wieland and Fischer used filter paper strips saturated with an acetate buffer as a medium for electrophoresis, while studying the separation of simple amino acids and of the complexes formed when various amino acids react with cupric ion (*14*). By incorporating radioactive copper into the complexes, cutting the strips into narrow bands and determining their radioactivity, they explored the possibility of using this method as a quantitative technique (*15*). To maintain the strips at constant temperature and lessen the effects of heating, Cremer and Tiselius used filter paper strips between glass plates and immersed in a bath of chlorobenzene (*16*). They separated the protein fractions of normal serum and showed that they correspond to those obtained in the classical Tiselius apparatus. In the author's laboratory, electromigration on paper and other materials, has been employed to study the feasibility of determining mobilities and transference numbers of inorganic ions (*2*), to analyze amino acid mixtures, to fractionate complex protein samples such as an ACTH preparation (*17*), and to determine isoelectric points (*18, 19*).

Durrum, using paper ribbon and sheets draped symmetrically over a frame-work so that their apex is elevated above the ends, which dip into the buffer solution, achieved a good separation of amino acids and a fractionation of serum proteins (*6*). Separations of serum proteins have also been carried out by Grassmann, Hannig, and Knedel (*20*). Kraus and Smith used electromigration on filter paper to study the sign of the charge of inorganic complex ions and for obtaining estimates of the stability constants of such complexes

Figure 2. An Ionograph in Unitized Form, with Line-operated Rectifier, Water-jacketed Chamber, Levelling Screws on Base, Adjustable Frame with Built-in Spirit Level. The Electrode Vessels Are at the Front

(*21*). Further articles which contain reports on the use of filter paper for electrophoretic studies include Biserte (*22*), Turba and Enenkel (*23*), Schwarz (*24*), and Knedel (*25*). Knedel reports a novel treatment of the paper, after color development, with a mixture of liquid petrolatum and α-bromonaphthalene. This treatment renders the strip transparent, so that the optical density of the colored regions can readily be determined on a transmission densitometer.

More recently, Kunkel and Tiselius have described an arrangement in which one or more filter paper sheets are clamped between glass plates and the edges sealed with silicone grease to prevent evaporation (*26*). Under such conditions, where the migrant is forced to travel through the body of the paper itself, they report that the factor of electroosmosis and the character of the individual paper used, play important roles. Flynn and de Mayo have combined certain features of Durrum's apparatus (*6*) and that of Cremer and Tiselius (*16*) to yield a qualitative method suitable for routine clinical work (*27*). They report that when the paper is held between glass sheets the advantage of control of evaporation was offset by interferring surface tension and capillary effects, and by a loss of protein on the glass.

In Figure 2 is shown a photograph of the instrument in current use in this laboratory.[1] In Figure 3 are shown some representative sketches of ionograms obtained in the separation of amino acid mixtures, by the technique outlined in this paper.

Factors Influencing Mobility. In order to utilize ionography to its fullest extent, it is necessary to know what factors influence the electromigration on paper and the character and range of these influences. Some of the fundamental aspects of the technique include the relationships that exist between the movement of the migrant and such factors as time, potential gradient,

[1] An improved form of the ionograph is available from Precision Scientific Co., 3737 Cortland Street, Chicago 47, Illinois.

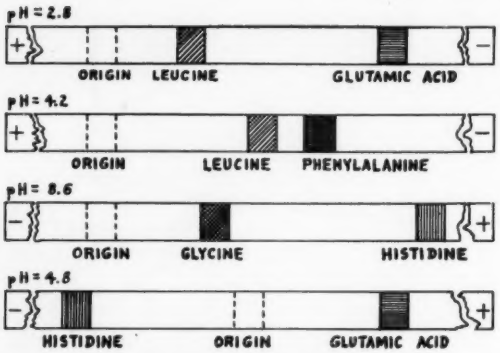

Figure 3.
Schematic Representation of Some Typical Ionograms Obtained from Amino Acid Separations

The pH listed is that of the buffer solution used. The upper two ionograms show the separation of two migrants, moving toward the negative electrode. The third shows the separation of two fractions moving toward the positive pole. The last illustrates a case where the pH of the buffer falls between the isoelectric points of the two amino acids, and they migrate in opposite directions on the paper.

pH of the buffer solution and its ionic strength, temperature, adsorption, and the role of electroosmosis.

As mentioned earlier in this paper a linear relationship between the movement of the migrating substance and time is observed, if precautions are taken to remove chromatographic and other possible interfering effects (*7*). It has also been shown that the rate of electromigration is directly proportional to the applied voltage

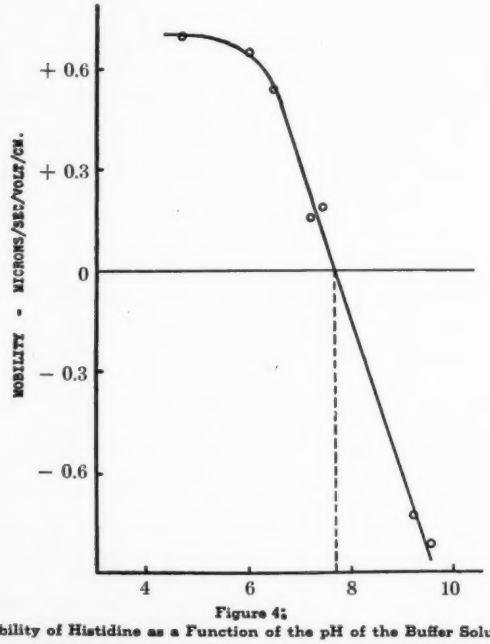

Figure 4.
Mobility of Histidine as a Function of the pH of the Buffer Solution

Potential Gradient, 15.7 v./cm.; Ionic strength, 0.013; buffers: pH 2.2–3.8, potassium hydrogen phthalate-HCl; pH 4.0–6.2, phthalate-NaOH; pH 5.8–8.0, potassium dihydrogen phosphate-NaOH; pH 7.8–10.0, KCl-NaOH; temp., 23–25°C.; time 3 hrs. The vertical broken line represents the isoelectric pH, 7.6.

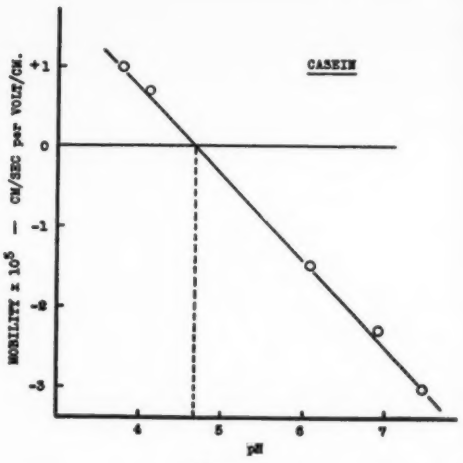

Figure 5.
Mobility of Casein as a Function of the pH of the Buffer Solution

Potential gradient 18.5 v./cm.; ionic strength, 0.0029; buffer, sodium diethyl barbiturate-sodium acetate-hydrochloric acid; temp., 23–24°C.; time, 4–16 hrs. The vertical broken line represents the isoleectric pH, 4.7.

(*19*). Under these conditions, the size and geometry of a particular apparatus ceases to be a factor of importance, and hence any convenient length of paper as well as any desired potential gradient may be used, as long as the movement is expressed in terms of mobility, *i. e.*, microns/second per volt/cm.

The pH of the buffer solution saturating the paper strip has a profound influence on the mobility of the migrant, particularly for ampholytes such as amino acids and proteins. The reactions of acids and bases with aqueous solutions of the latter substances can be presented schematically as follows:

$$NH_2\text{-}R\text{-}COO^- \xleftarrow{OH^-} {}^+NH_3\text{-}R\text{-}COO^- \xrightarrow{H^+} {}^+NH_3\text{-}R\text{-}COOH$$

If the mobility is measured in several buffers of constant ionic strength but varying pH, it is found that at some definite pH (the isoelectric point), the mobility of the amino acid or protein, and therefore its net charge, is zero.

At pH values of the buffer lower than the isoelectric point of the ampholyte (*i. e.*, on the acid side of the pI), the migrant bears a net positive charge and will move toward the negative end of the paper strip. At pH values higher than the pI (*i. e.*, on the basic side of the isoelectric point), the migrant bears a net negative charge and moves towards the positive end of the paper.

The isoelectric points of several amino acids and proteins have been determined by ionography and found to be in reasonably good agreement with the values determined by classical methods (*18*, *19*). In Figures 4 and 5 are shown the behavior of histidine and the behavior of casein respectively, the mobility

[2] Supplied through the courtesy of Armour Laboratories, Chicago.

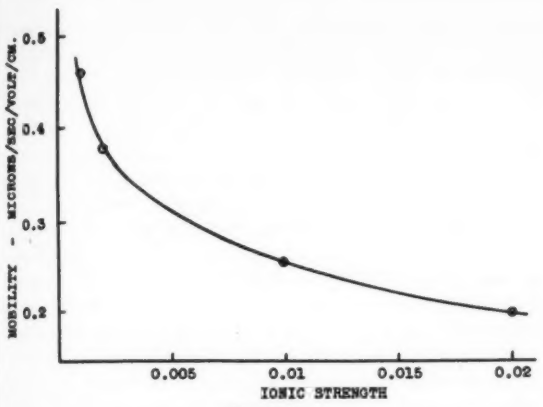

Figure 6.
The Mobility of Leucine as a Function of the Ionic Strength of the Buffer Solution

Potential gradient, 15.7 v./cm.; pH, 4.2; buffer, potassium hydrogen phthalate-NaOH; temp., 23–24°C.; time, 3 hr.

of positively charged ions being plotted above, and of negatively charged ions below the line corresponding to zero mobility. The isoelectric points of histidine and also of casein, as determined by ionography, are 7.6 and 4.7 respectively. Each point on the curves in Figures 4 and 5 represents the average of triplicate determinations. Displacements were usually about 12 cm., and were reproducible to the extent of ±2 mm.

When the ionic strength of the buffer solution is varied, while all other factors influencing the mobility are fixed, it is found that the velocity of migration is markedly altered. The mobility of the migrant increases with decreasing ionic strength in such a way that the rate of increase of migration velocity becomes greater as the ionic strength is lowered. In Figure 6 the mobility of leucine is plotted as a function of the ionic strength of the buffer solution. The observed behavior has a logical basis in the known effects of electrolyte concentration on the ionic atmosphere of a charged particle in solution. It is evident that the time required for separation of a mixture of components is less at lower than at higher ionic strengths of the buffer solution used to saturate the paper.

When the mobility is plotted against the reciprocal of the square root of the ionic strength, a straight line relationship is obtained over a considerable ionic strength range (*19*). This may be interpreted to mean that the migrant moves as if it were deep in the body of the liquid. An explanation for this behavior may be that the movement of the migrant occurs primarily in the water sheath surrounding the filter paper strip. This possibility would explain the observation that the mobility of bovine serum albumin and other substances was observed to be markedly reduced when the filter paper strips are wetted with less than the usual amount of buffer solution.

A priori, there would appear to be some justification for assuming that the results obtained by ionography might be somewhat different from those obtained by free electrophoresis, because of the effect of electroosmosis. If electroosmosis were appreciable, it has been pointed out that the mobility of positively charged ions might be expected to be increased, while that of negatively charged ions might be decreased (*2*). This view is based on the consideration that, in general, the paper surface appears to bear a negative charge, while the water acts as if it were positively charged. Consequently, the resultant velocity vector of the positive ions would be expected to be the *sum* of the electrophoretic velocity and the electroosmotic velocity of the water in which they are being bouyed along. Negative ions, on the other hand, might be pictured as breasting the electroosmotic tide. The resultant velocity would then be the vector *difference* between the electrophoretic velocity and the electroosmotic velocity of the water.

If this presentation reflects the true state of affairs during electromigration on paper it would be expected that, in the case of an hypothetical ampholyte, the free electrophoretic mobilities would be represented by line *A* (Figure 7), while the mobilities on paper would be represented by line *B*. It would then follow that the isoelectric points determined by ionography would be higher than those obtained by other methods. The magnitude of the difference in the isoelectric points should be a measure of the amount of electroosmosis.

The data reported herein indicate that essentially the same isoelectric points are obtained by ionography as are obtained by other methods. In another laboratory Schwarz (*24*) has recently determined the isoelectric point of horse serum albumin by electrophoresis on paper, and found it to be 4.76. Data of Tiselius, (quoted by Schwarz) obtained on the same substance by the moving-boundary method, indicated that the isoelectric point was 4.64. The differences in these two measurements, which is less than 3 per cent, could well be within experimental error. The effect of electroosmosis would therefore appear to be a relatively negli-

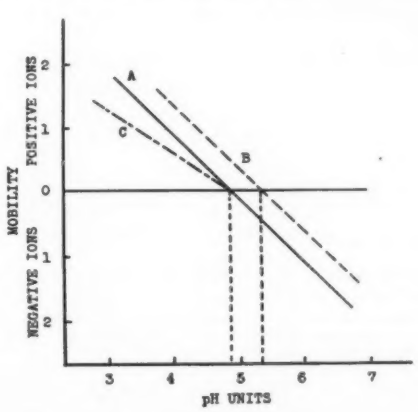

Figure 7.
An Idealized Schematic Representation of the Effects of Electroosmosis and Adsorption on the Mobility of Positive and Negative Ions

A, an hypothetical curve for an ampholyte in the absence of electroosmosis and adsorption; *B*, effect to be expected from electroosmosis; *C*, the broken line and that portion of line *A* below the zero mobility axis illustrate the effect to be expected due to adsorption.

gible factor in ionographic measurements, when conducted as described here.

Further evidence in support of this view is furnished by the fact that the mobility of crystalline bovine serum albumin was found to be the same when determined on woven glass-fiber ribbon ("Fiberglas" woven tape, continuous filament, tightweave, B; $^3/_8$ in. in width; 0.007 in. in thickness) and on filter paper, under otherwise identical conditions (*28*). This identity of results would not be expected unless a relatively free pathway were available through which the migrant could move. Because of the difference in surface-charges at the glass-buffer and cellulose-buffer interfaces, the mobility of the migrant would be expected to be different, if it moved within even a few molecular diameters of these interfaces.

These observations may then be interpreted to mean that, under optimal conditions the migrant may move at such a distance from the cellulose-buffer interface that the electroosmotic transfer of water through the paper does not appreciably affect the mobility of the migrant. It seems probable that techniques of electrophoresis on paper, in which the water sheath surrounding the paper is eliminated (by clamping between glass plates, etc.), might give results in which the effects of electroosmosis are magnified. However, even under these conditions, if it is desired to compare the results with those of the moving boundary method, it is easy enough to apply a correction factor to the mobilities. This is done simply by running, along with the unknown migrant, some reference material whose mobility, by the moving-boundary apparatus, is well established.

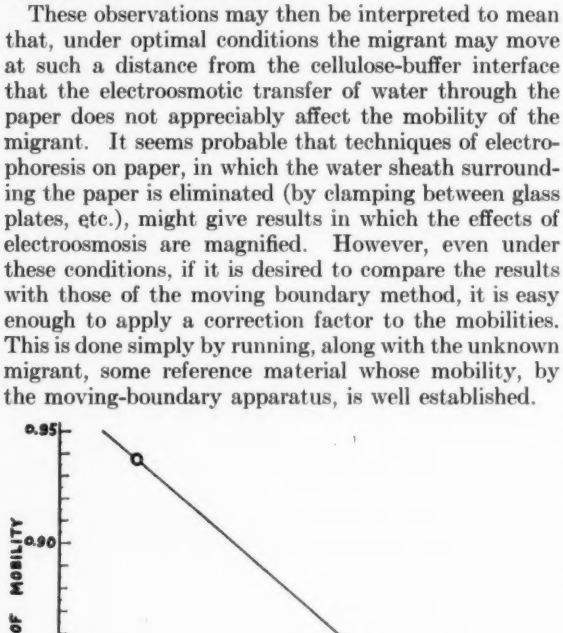

Figure 8.

Effect of Temperature on the Mobility of Bovine Serum Albumin

Open circles, determined by ionography. Closed circle, determined by moving boundary; mobility, 6.66 microns/sec. per volt/cm. at 274°K. Mobility by ionography at 274°K., 6.50. Veronal buffer; ionic strength, 0.1; pH, 8.6; helium atmosphere.

In Figure 8 is shown the relation of mobility to temperature, for bovine serum albumin. One of the points on this graph was determined on an aliquot of the same sample by the moving boundary method.[3] The experimental conditions such as type of buffer, ionic strength, and pH were identical. It is evident that the mobility of this particular substance as computed from the ionographic method agrees within experimental error with that obtained from the moving boundary method.

It should, perhaps, be stated that whether or not the mobilities obtained by ionography agree exactly, in all cases, with those obtained by the moving-boundary technique is not really too important. What is important is that the data obtained from ionographic measurements be reproducible. At the rate at which the technique is being explored and adopted in various laboratories it will not be long before a rich deposit of experimental data is available in the literature and the discipline of ionography can stand on its own merit, without need of reference, for corroborative purposes, to other methods of electrophoresis.

It is not unreasonable to expect that different types of filter paper would exhibit different amounts of adsorption toward a migrant. Therefore, a series of experiments was carried out using a variety of filter papers to determine whether any difference in mobility of the migrant (bovine serum albumin) could be observed which might be attributed to the nature of the paper surface. Six varieties of Eaton and Dikeman paper, differing in thickness, rapidity of filtration, smoothness of surface, and wet strength were employed. It was found that the mobility of the migrant was the same on all six types of paper (*28*); hence, it may be concluded that the character of the paper itself does not introduce any important variables into the measurements, at least when the migrant is negatively charged, and when the experiments are carried out in the manner described here. It appears, however, that if the paper strips or sheets are clamped between glass sheets, the migrant is forced to travel largely through the body of the paper rather than through the surrounding liquid sheath, and the geometry of the individual paper then becomes a factor which must be contended with (*26*).

The possible effect of adsorption of proteins and amino acids on paper, on their electrophoretic mobilities, can be delineated as follows. It might be expected that due to the slight negative charge on the paper surface, the electrophoretic velocity of positive ions would be decreased, while that of negative ions would be relatively unaffected. Thus, the factor of adsorption, by itself, would not be expected to cause any appreciable shift in the experimentally determined isoelectric points of amino acids or proteins. It would be expected, in general, that the lower the pH of the buffer solution below that of the isoelectric point of the migrant, the greater would be the adsorption of the latter. As the pH of the buffer solution approaches the pI of the migrant, however, the effect of adsorption would be expected to decrease to zero. As a rule it would seem preferable to run ionographic determinations on ampholytes in a buffer whose pH was greater than that of the pI of the migrant. Under such conditions the factor of adsorption would be minimal. It should be stated that

[3] This sample of protein has been used as a standard in electrophoretic experiments in Armour Laboratories, and has been checked in several other laboratories.

amino acids and proteins so far studied, are readily washed from the paper, a fact which in itself supports the contention that for such substances the adsorption on the paper may not be a serious interfering factor.

An investigation was made of the effect of the concentration of the migrant itself on the widening of the zone produced as it moved on the paper strip under otherwise standardized conditions. For this purpose, six solutions of glycine were made up covering a concentration range from 0.027 to 2.680 molar. A constant volume of the various solutions was added to individual paper strips in an amount sufficient to produce on spreading an initial zone 1.2 cm. in width. The zones were then made to migrate on the paper strips for three hours, during which time all moved a uniform distance of 8.4 cm. The following conditions were maintained during the experiment: potential gradient, 8.05 v./cm.; pH=8.6; ionic strength, 0.10; buffer, veronal; temperature, 24°C.; helium atmosphere. It was found that the mobilities of all samples were identical and that the width of the migrant zones remained unaltered despite the large relative differences in the concentration of the migrant. In other words, the zone moved as an electrophoretic unit.

SOME APPLICATIONS

As a method of achieving electrophoretic separations and for determining mobilities, ionography combines many of the better features of the older methods for electrophoresis with some novel inherent advantages of its own. Perhaps the most striking aspect of the technique is that it lends itself equally well to the handling of small ions such as amino acids, and of larger particles, such as colloidally dispersed proteins. Another important feature is the fact that since convection currents are of no great consequence in ionography separations may be carried out at any desired temperature, and the mobility of the migrant, as well as its temperature coefficient may be determined readily.

In the past, when electrophoretic measurements in aqueous media were made at temperatures other than in the neighborhood of 4°C., the point of maximum density, convection currents offered a serious difficulty. As very few nonaqueous liquids or liquid mixtures exhibit points of maximum density, the study of electrophoresis was restricted largely to water solutions or suspensions. In ionography the elimination of convection currents allows electrophoretic studies to be extended to solutions of water with certain other liquids, such as dioxane, butanol, etc. This feature is important in biochemical work, where some materials whose electrophoretic characteristics are of great interest, are soluble in water to only a very limited extent.

The amount of substance required in the ionographic method (a few micrograms) is small compared to that required in the moving-boundary method. Again the measurements on paper have been extended down to a buffer ionic strength of 0.001 without untoward difficulty, while in the moving-boundary method it has been suggested that it is inadvisable to work at buffer ionic strengths less than 0.02 (*29*). This may be important in certain cases since the rapidity of resolution of components having nearly identical mobilities is increased at lower ionic strengths.

Since resolution in electrophoresis is a function of the field strength it is desirable to use as large a potential gradient as possible without causing convection, in the older methods, or without too great an evaporation rate, in ionography. In any case, the maximum feasible potential gradient is an inverse function of the specific conductivity of the solution. Hence greater potential gradients can be employed with buffers of lower ionic strength, and consequently ionography has, in this respect, an additional advantage.

The technique can be employed to determine the electrophoretic homogeneity or heterogeneity of the dissolved or dispersed phase in an unknown solution; for example, it has been used to separate and identify the individual constituents in mixtures of amino acids and proteins. The mobilities of the components can be determined readily under a wide range of conditions, such as variable pH, ionic strength, temperature and buffer type, and therefore a study of relationships existing between the mobility and these controllable factors is simplified. Isoelectric points of amino acids and proteins have been determined by ionography (*18*, *19*, *24*).

Complete separation of the constituent fractions of a mixture, such as amino acids or proteins, can be achieved, rather than just a separation into concentration gradients. The technique may therefore be employed for preparative purposes. The zone of the paper strip containing a particular fraction can be cut and the migrant eluted by customary methods. Thus isolated, its quantity or specific biological action can be determined. This sort of separation, achieved without chemical reaction or modification of the material, and even when the nature of the constituents is unknown, is particularly important with physiologically active materials (*17*, *30*). Another variation in the technique is to replace the several paper strips by one wide paper sheet, which is used in conjunction with a buffer of low ionic strength. A larger quantity of the migrant can be handled and a particular fraction run off the paper directly into the buffer vessel, from which it may be recovered by low temperature evaporation or other suitable procedures.

Since the method lends itself to the determination of ion mobilities it may be employed to study complex-ion formation in solution (*21*) and under certain limited conditions to determine transference numbers for inorganic electrolytes (*2*). It may be valuable as a supplementary method for the micro qualitative analysis of inorganic ions. Preliminary experiments indicate that a solution of several such ions can be fractionated, when run for a sufficiently long time. Simple spraying of the dried strip with a solution of ammonium polysulfide, for instance, develops satisfactory colored zones with many inorganic ions except those of the alkali and alkaline earth groups. In some cases one or more ions,

whose mobilities are nearly alike, can be separated by forming complexes of some of them and thus altering their relative mobility or even their direction of movement. At the proper concentration of chloride ion in the solution, for example, lead, mercuric, and bismuth ions may exist as negatively charged complexes and migrate toward the positive pole.

Two dimensional experiments are easily carried out. A square sheet of paper is used, and the migrant is applied at one corner of the sheet. It is customary to run it in the first direction using a buffer of low ionic strength and definite pH. After some definite time, the paper is rotated through 90 degrees and set back in the ionograph, using a buffer of the same kind, but of a greater ionic strength, and either the same or different pH.

One value of this technique has been to show that the trailing areas often observed in one-dimensional paper electrophoresis may represent a host of minor constituents with varying mobilities. When a zone, with its trailing area, migrates in the second direction, it is sometimes observed that it becomes arranged in a diagonal pattern, *i. e.*, particles at the forward and rear end of the band, from the first direction, move at different rates in the second direction. This behavior can be interpreted to mean that the zone is not homogeneous (*26*). It must be made up of several components whose mobilities are very close together. An attempt is made to illustrate this point in Figure 9.

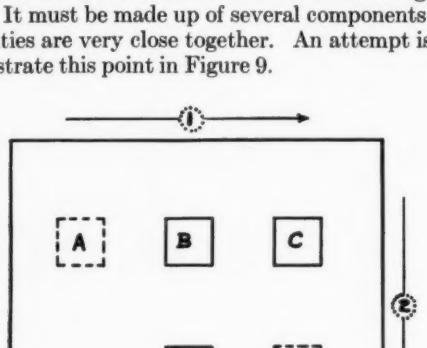

Figure 9.
An Idealized Representation of a Two-Dimensional Ionogram

The original area of application, *A*, breaks up into 2 zones, *B* and *C*. After electromigration in the second direction, the shape of zone *B* is relatively unchanged, indicating homogeneity, while zone *C* undergoes a diagonal shift, indicating that it is not homogeneous and contains materials having a range of mobilities.

The ionograph itself is readily portable and it is well to remember that several strips, with many applications of the migrant per strip, can be handled simultaneously, thus affording a convenient check on a given separation procedure. The dried paper strips, or ionograms, furnish permanent records of an experiment, and the important sections can be excised and pasted directly into a laboratory notebook for later reference. It appears from preliminary experiments that certain proteins may be stained with suitable dyes, or their functional groups altered in other ways, thus providing them with new mobility characteristics and permitting a separation from other proteins where the native mobilities are very closely alike. Preliminary work, in this laboratory, has shown that it is possible to record the results of electromigration on a strip-chart recorder.

It is true that some experience with the apparatus is necessary to become acquainted with the many minor difficulties that beset the way of the novice. However, it can be expected that radical advances in the apparatus itself, in the handling of the ionograms, and in ingenious applications will be made. This new frontier in electrophoresis offers every promise of developing into a major analytical and research advance.

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THE IDENTIFICATION OF ZINC IN THE PRESENCE OF NICKEL AND COBALT

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For the past few years in our classes in inorganic qualitative analysis, we have been using a procedure,[1] according to which zinc, nickel, and cobalt are separated as the ammonia complexes. These are converted to the sulfides, and finally dissolved in nitric acid. Zinc is separated from nickel and cobalt by removing the latter as $Ni(OH)_2$ and $Co(OH)_3$ in excess sodium hydroxide. The zinc is then present as $Zn(OH)_4^{--}$ and is isolated by centrifuging and removing it from the precipitated hydroxides. The zinc is identified by treatment with H_2S, which yields a white precipitate of ZnS.

Two major difficulties have presented themselves in the course of this procedure. The treatment of $Zn(OH)_4^{--}$ with H_2S very often results in a black precipitate which is undoubtedly due to the incomplete separation of nickel and cobalt. Furthermore, in the presence of nickel and cobalt small amounts of zinc cannot be isolated and identified.

It was thought that the presence of NH_4^+ caused the incomplete precipitation of $Ni(OH)_2$ and $Co(OH)_3$. In alkaline solution, free ammonia would be formed which would tie up nickel and cobalt as the soluble ammino complexes, $Ni(NH_3)_6^{++}$ and $Co(NH_3)_6^{+++}$ and prevents their complete precipitation as hydroxides and subsequently their removal from zinc.

On analysis of solutions containing Zn^{++}, Ni^{++}, and Co^{++}, in concentrations varying from 0.5 mg./ml. of zinc, nickel, and cobalt to 0.5 mg./ml. of zinc and 5 mg./ml. each of nickel and cobalt, it was found that centrifuging does not afford a satisfactory separation of nickel and cobalt hydroxides from the zincate ion. The centrifugate if treated with H_2S gives a decided black precipitate. However, if the centrifugate is filtered through a standard filter paper and then treated with H_2S, a clean, white precipitate of ZnS is obtained. In spite of the fact that the centrifugate appears clear, finely suspended particles of nickel and cobalt hydroxides are undoubtedly present.

Pure zinc sulfide is precipitated by this procedure even in the presence of 50 mg./ml. of NH_4Cl, showing that Ni^{++} and Co^{+++} were not kept in the solution as the ammino complexes, $Ni(NH_3)_6^{++}$ and $Co(NH_3)_6^{+++}$, as was previously supposed, but rather mechanically, and as such can be removed by careful filtration.

The fact that the presence of nickel and cobalt interfere with the identification of small amounts of zinc leads one to believe that zincate ion, $Zn(OH)_4^{++}$ is adsorbed by the nickel and cobaltic hydroxides. To confirm this, zinc in combination with various amounts of nickel and cobalt was separated by the methods described above, and the amounts of zinc remaining in solution were then determined quantitatively by titration with potassium ferrocyanide.[2] The results of these determinations are summarized in the table, which shows that the presence of large amounts of nickel and cobalt makes the determination of zinc less reliable and a semiquantitative measure of the amount of zinc less accurate. Since students are required to report the amounts of metals present in their unknowns, it is pedagogically unwise to give them traces (less than 1 mg./ml.) of zinc together with large amounts (more than 5 mg./ml.) of nickel and cobalt.

Zn^{++} added, mg./ml.	*Co^{++}, mg./ml.*	*Ni^{++}, mg./ml.*	*Zn^{++} found, mg./ml.*
1.57	..	..	1.57
1.57	1	..	1.26
1.57	2	..	1.01
1.57	4	..	1.10
1.57	8	..	0.62
1.57	10	..	0.66
1.57	..	1	1.38
1.57	..	2	1.21
1.57	..	4	0.18
1.57	..	8	0.86
1.57	..	10	0.29

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THE "COMPLEX" CHEMISTRY OF IRON

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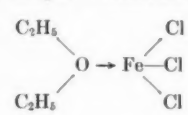
THE rapidly expanding body of knowledge dealing with the chemistry of the transition elements requires not only continuing integration, but also an analysis of the current problems on which present theory has become snagged. The example of iron is an excellent case in point. The ferric ion, along with the cobaltic ion, possesses one of the highest charge:diameter ratios in the transition series, although prediction of complexing ability or other properties from this basis alone is rather uncertain (*1*). A complete explanation of all problems to be detailed below would require a thorough knowledge of all forces operating at the molecular level, and a valid, all-inclusive theory of valence—conditions that are still only partially satisfied.

Conflicting statements and sometimes contradictory experimental data impair the effectiveness of a survey; however, if it is realized that to a large extent these contradictions result from the interaction of the system under study with its environment, then seemingly contradictory evidence may at least be systematized. Thus, as pointed out by Wells (*2*), ferric chloride exists as a monomer in the vapor phase at high temperatures, and an infinite two-dimensional molecule in the anhydrous crystalline state, whereas upon solution of the salt, the products depend almost entirely upon the nature of the solvent. Inasmuch as it is not very often possible to identify or even qualitatively describe the sum total of all factors governing molecule formation, it is believed to be necessary to adopt a broad viewpoint as to what constitutes a molecule. In this respect, the authors follow the definition of Glockler (*3*) that a molecule is "any configuration of two or more atoms, simple molecules or radicals which shows a potential minimum." On this basis it is not possible to sort out the various distance-dependent electrical forces into valency, van der Waals, or dispersion forces for a molecule intermediate in binding between the helium molecule held together by van der Waals forces only, and the covalency of the hydrogen molecule. Instead, a transitional scale must be set up between the extremes, within which the various forces merge and overlap in characteristics. Within this transitional framework, however, the usual descriptive terms may be arbitrarily used.

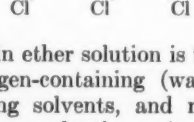
The problems of correlation of crystal structure with interatomic forces have been more successfully treated, from the viewpoint of quantitative data, than the corresponding questions of structure of solution, and excellent summaries of our knowledge in regard to the nature of the solid state are available in standard treatises (*4*, *5*, *6*). In many respects, study of solutions of complex ions is more fruitful as regards dependence of bond type on the nature of the complexed group, since it is often possible to preserve the general form of the crystal complex by proper choice of the solvent. Since the major portion of the unresolved problems of iron chemistry are concerned with the interaction of its compounds with solvent systems, this review will be limited primarily to this area.

SOLVATION

One of the principal sources of confusion in the treatment of solutions of a crystalline salt has been lack of full realization that the process of solution is rarely reversible with respect to the initial formulation. While solution of the Fe_2Cd_6 dimer in a nonpolar solvent like CS_2 will yield the original salt on evaporation, solution in polar solvents will yield diverse products entirely dependent on the nature of the solvent. Thus, in ether solution, simple tetrahedral molecules,



are formed, in which the tetrahedral arrangement of the initial four iron bonds in Fe_2Cl_6 is preserved (*7*):



This behavior in ether solution is typical of solution reactions in oxygen-containing (water excepted) or nitrogen-containing solvents, and may be described by the generic term, solvation. An interesting extension of this class of reaction is the tendency of iron compounds to form addition products with cellulose. Nikishin (*8*) found that the addition of iron compounds to sulfite cellulose extract resulted in complex bonds so stable that the typical ionic reactions of Fe^{+3} were not obtained. It is quite possible that this may be a partial explanation of the traditional place of iron in permanent aqueous writing fluids, although little work

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appears to have been done on this problem. The most familiar type of solvation is, of course, hydration, but the complexity of this common process has been fully examined only within recent years.

Bjerrum (*9*) treated the formation of aquo-ions as a statistical problem in stepwise complex formation with suitable modification for steric effects and previous association products. Although formulations were based originally on experimental work on metal-ammine solutions, Bjerrum extended them to aquo-ions on the assumption of complete interchangeability, indicating definite and one-to-one correspondence for the aquo-complex. Complex formation was found to be more marked than could be accounted for on the basis of charge density, and the bonds of the resulting complex were highly directional in character. Bjerrum postulated hexaquo octahedral iron-group ions, but from a consideration of the ionization energies involved, it is certain that the ferric ion could not exist as such in solution without extremely high energies of hydration or equivalent coordination.

COMPLEXITY OF ION COMPOSITION

It is now known that solution of a simple ferric halide in water gives rise to a whole host of products ranging all the way from the completely hydrated free ions to the most electronegative anionic specie, FeX_6^{-3}, with all intermediate association products present. Since a definite concentration of OH^- ions is always present, it is to be expected that there will be mixed complexes involving the hydroxyl group as a ligand. The formula of the ion-molecule must necessarily include the attached water molecules, if only by reason of bond tenacity, and it has recently become evident that their omission is a serious oversight in the formulation of reaction mechanisms. In the presence of the components of the solution, iron-aquo complexes generally give analytical tests for the simple ions, in addition to complex-ion indications, which relegates them to the lower end of the scale of complex stability. Thus, neglecting all *anionic* species the "simple" solution of $FeCl_3$ in water can be represented:

$$FeCl_3 + H_2O = \begin{cases} Fe(H_2O)_6^{+++} \\ Fe(H_2O)_5Cl^{++} \\ Fe(H_2O)_4Cl_2^{+} \\ Fe(H_2O)_3Cl_3 \\ Fe(H_2O)_3(OH)Cl_2 \end{cases} + \begin{cases} Fe(H_2O)_5(OH)^{++} \\ Fe(H_2O)_4(OH)_2^{+} \\ Fe(H_2O)_3(OH)_3 \\ Fe(H_2O)_4(OH)Cl^{+} \\ Fe(H_2O)_3(OH)_2Cl \end{cases}$$

It is true that the concentration of most of the products listed above will be negligible, but in the spectral methods generally used for identifying ions, even "negligible" concentrations may be detectable. Obviously, adjusting the pH to suppress the concentration of OH^- only serves to introduce still another associating ion, unless a noncomplexing anion is used. Perchloric acid is commonly used for suppression of the OH^- concentration, although it has been observed that even at concentrations of 3 *M*, association with OH^- is still sufficient to interfere with spectral results (*10*). One of the dangers of such a procedure has been demonstrated by Nekrasov and Ovsyankina (*11*) who in work on phase relations and distribution in the system $FeCl_3$–HCl–H_2O–Et_2O established the communicating complex across the phase boundaries as an acid containing more than one anion.

Inasmuch as most studies of ferric complex formation in solution have had perforce to deal with two salts; *i. e.*, three or four ionic species, in addition to those already present in water, special techniques have been required for the isolation of the particular ion-molecule under investigation. Bent and French (*12*) and Edmonds and Birnbaum (*13*) in duplicate photometric studies of the equilibrium involved in a ferric thiocyanate solution concluded that the source of the red color was the $Fe(SCN)^{++}$ ion (actually the $Fe(H_2O)_5SCN^{++}$ ion-molecule) and were able to calculate the first association constant for this product. Utilizing the colored ionic complex as the indicator, Bent and French were also able to determine the first association constant of Fe^{+++} with Cl^-.

FERRIC THIOCYANATE SOLUTIONS

On the other hand, Babko (*14*) conducted similar spectrophotometric studies on ferric thiocyanate solutions and interpreted his extinction data in terms of "stepwise complex formation" by assuming that six complexes ranging from $Fe(SCN)^{++}$ to $Fe(SCN)_6^{-3}$ coexist in varying proportions. Babko also obtained confirming data from electrolytic transference determination of his solutions. With a Fe^{+3} concentration of 0.002 mol per liter and a SCN^- concentration of 0.01–0.02 mol per liter, he found that the colored complex layer moved toward the cathode; at concentrations of SCN^- above 0.2 mol per liter, the motion was reversed, and the colored layer moved toward the anode. This investigator also found that Beer's law did not hold on dilution with water, since such dilution was accompanied by a change in concentration of the coordinated complex ion; it holds only if the SCN^- concentration is maintained as the Fe^{+3} concentration is decreased by dilution. In this latter instance, the role of the water ligands is readily apparent; assuming a dynamic equilibrium, and participation of H_2O molecules in the reaction, consideration of the law of mass action leads directly to this observed effect.

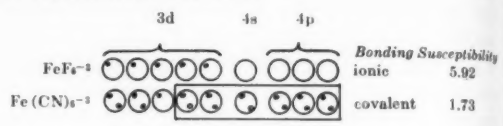
Corroboration of higher association complexes of SCN^-, including anionic species, has been obtained by Möller (*15*), by Schlesinger (*16*), and by von Kiss, Abraham, and Hegedüs (*17*). In view of the evidence for the existence of higher association products, even in dilute solutions, Rabinowitch and Stockmayer (*10*) point out that "the color of a given ferric salt solution can be due to any or all of a multitude of associated products, and only a detailed investigation of the spectrum at different concentrations, acidities, and temperatures can give some indication as to the true origin of the absorption bands." Rabinowitch and Stockmayer, employing automatic spectrophotometry, successfully

disengaged the spectrum of the Fe^{+3} ion from its association products, and by carefully controlling solution variables were able to determine the spectra of a group of iron halide complexes: $FeCl^{++}$, $FeCl_2^{+}$, $FeCl_3$, $FeBr^{++}$, and $Fe(OH)^{++}$. Equilibrium constants and heats of formation and entropy were evaluated by suitable temperature and concentration-variation methods for these complexes. While this paper is a comprehensive piece of work, there nevertheless appear to be curious anomalies in the observations. For example, it was observed that both for $Fe(OH)^{++}$ and $FeCl^{++}$ the yellow color was enhanced by an increase in temperature, yet heats of formation derived from the effect of temperature on the equilibrium constant of association indicated that the association with OH^- ions is exothermal, and that with halogen ions, endothermal. If, as the investigators declare, the electrostatic energy gained by the association of Fe^{+3} with the halogen anions does not compensate for the loss of hydration energy, the reverse must be true for the $Fe(OH)^{++}$ complex, and on the basis of the thermodynamic data one would therefore expect a color fading with temperature increase.

The authors of this review, in work on pure ferric thiocyanate solutions, have observed a marked color fading with temperature increase, consistent with Rabinowitch and Stockmayer's findings for the affinity order of Fe^{+3}: SCN^- OH^- X^-, but a fact in contradiction to the color deepening of $Fe(OH)^{++}$ with increasing temperature. There would appear to be adequate grounds here for suspecting an unexpectedly large variation in the strength of the Fe^{+3}—H_2O ion-dipole bonds with substitution in the hydration sphere. Certainly, an investigation of the reverse phenomenon, that of replacement of a negative constituent in the hydration layer with a water molecule, would serve to check this point. Of particular interest is the question of reversibility of the thermally susceptible colored complex. It may be mentioned here that the phenomena described so far are by no means limited to the transition elements, but have been observed for complexes of Bi and Pb (*18*, *19*).

MAGNETIC SUSCEPTIBILITY

The most widely accepted criterion for distinguishing between ionic and covalent types of binding in complexes of the transition group is the measurement of the magnetic susceptibility. Extensive measurements have been carried out on complex compounds and their aqueous solutions and the pertinent data have been summarized by Pauling (*20*). Since the magnetic moment resides in the transition–group element itself, ionic bonding leaves the ionic moment of Fe^{+3} unchanged, whereas covalent bonding reduces the moment by coupling electron moments. By the Hund multiplicity rule, Fe^{+3} with five electrons in the *3d* orbitals has a magnetic moment of 5.92 Bohr magnetons, whereas Fe^{++} with an additional electron has only four unpaired electrons and susceptibility of 4.90 magnetons. Thus, the electron distribution in the orbitals for FeF_6^{-3} can be represented as follows:



The filling of the orbitals is governed by the rules developed by Pauling (*21*) on the basis of quantum treatment of the directive properties of covalent bonding; this treatment does not apply to ionic bonding or forced configurations. In the octahedral complexes of iron the hybridization of the d^2sp^3 orbitals is involved. Observations that the susceptibility is that corresponding to the ionic state are not necessarily conclusive, inasmuch as weak covalent bonds could be formed by utilization of the *4s* and *4p* orbitals, leaving the *3d* orbitals undisturbed. However, an sp^3 hybridization corresponds to a tetrahedral distribution, a distribution not observed as yet for iron complexes.

In recent work on the magnetic moments of iron complexes, Werbel, Dibeler, and Vosburgh (*22*) found no change in moment for a ferric perchlorate solution upon introduction of oxalate, chloride, or thiocyanate ion. A decrease in moment was observed with addition of acetates, lactate, tartrate, citrate, and phosphate ions. For these latter cases, the moment was found to be a function of the excess anion concentration and the acidity, and these data were interpreted as an indication of stepwise complex formation. An adequate theoretical explanation of this difference in anionic behavior has not been advanced.

The possibility of low-temperature stabilization of the apparently covalent type of linkage in iron complexes has been intimated by several investigators. Klemm and Raddatz (*23*) obtained clear evidence of this phenomenon by using salicylaldehyde-ethylenediimine as a complexing agent in solutions of $FeCl_3$. The value of the magnetic moment was found to decrease with decreasing temperature, and this temperature dependence was explained by postulating the formation of a planar complex at the lower temperatures. This information parallels the thermally sensitive behavior of the colored ferric salts mentioned earlier, salts whose aqueous complexes are supposedly of a "pure" ionic type. The dual nature of the electrostatic coordination of the aquo-ions has been pointed out by Emeléus and Anderson (*24*). They state that "the purely electrostatic binding probably represents only a limiting type, and it is likely that the coordinate link is in many cases a bond of 'fractional order,' intermediate between a purely physical and a true chemical linkage."

ELECTRON TRANSFER

Even though the complex be ionic, some of the characteristic features of covalent binding, such as directive tendencies, may arise from the quite different

causes of steric effects and symmetrical space distribution of competing anions. Recent work with radioisotopes has cast suspicion on the validity of some of the conclusions drawn from magnetic susceptibility measurements. Van Alten and Rice (*25*) measured the rapidity of electron exchange between ferric and ferrous ions in perchloric acid solution with radioferric ions and found the amazingly long half-time of exchange of some 20 days. Since magnetic moment measurements of the octahedral complex of iron with water indicate essentially ionic bonding, one would be led to expect almost immediate electron transfer. Libby (*26*) has advanced several possible explanations of this behavior; transfer may be retarded due to a difference in hydration spheres of the ferrous and ferric ion, or the exchange may possibly take place through an intermediate complex whose statistical probability is small. In connection with this, it is interesting to note that Van Alten and Rice observed that the diffusion of ferric ion was more rapid than ferrous ion.

Dain, Kutsaya, and Liberzon (*27*) explored the electron-affinity spectrum of the hydrated ferric ion and found that the long-wave limit of the spectrum coincides with the work of breaking away the electron from the dissolved ion. The primary act was postulated as electronic transition taking place within the complex of the ion-hydrate shell. It may be that the stability of the complex ion-hydrate is such that the solvation layer acts as a protective sheath, screening the core ion from activation by all but the severest collisions. On this basis, agents with the greatest complexing power should offer the best protection against oxidation. Exactly the opposite effect has been observed (*28*). Oxidation of ferrous chloride solutions by an air stream takes place very slowly; addition of a complexing agent, such as an acetate, greatly accelerates the rate of oxidation. The rate also shows stepwise dependence on the concentration of the complexing ion.

Adamson, Welker, and Volpe (*29*) measured the rates of exchange of radio-KCN with $K_3Fe(CN)_6$ and $K_4Fe(CN)_6$ and found negligible exchange in neutral and basic solutions, in agreement with the conclusions of covalency for these compounds arrived at from magnetic moment measurements. However, they also found that radiocyanide was oxidized in the dark by ferricyanide, without exchange, thus providing confirmation that the ferricyanide-ferrocyanide couple involves a direct electron transfer. These authors are of the opinion that their slow CN^- exchange results can be best explained on the basis of substitution reactions such as those leading to aquo complex formation, a mechanism that, as indicated above, undoubtedly merits further experimental attention. In discussing electron-transfer spectra, Rabinowitch (*30*) set forth the conditions conducive to the transfer of electron from one atom or ion to another. In order that such a transition shall occur, it was stipulated that the bonding forces must be relatively strong, yet not strong enough to interfere with the assignment of electrons to individual ligands. It is apparent that the bond types satisfying these conditions would have to be ionic or ion-dipole in nature; the covalent bond is definitely excluded.

EFFECT OF pH

The effect of pH on the mechanism of electron transfer has been directly measured by Lingane (*31*) whose polarographic data indicated that the ferric-ferrous couple was reversible only at pH values smaller than 7. Here again, the role of stepwise formation of complexes of intermediate stability is implied, inasmuch as the reduction wave for the ferric-ferrous transition in tartrate media divides into two separate waves for a pH range of 6.2 to 8. Lingane attributes this to the formation of two different iron complexes, involving the hydrogen tartrate ion and the tartrate ion. The part played by stepwise acid-base reactions resulting in products of the form $H[Fe(H_2O)_2X_4]$ or of $H(Fe(FeX_6)]$ must also be considered; Babko (*32*) estimated that the energy of the chemical bonding of the coordinating anions with Fe^{+3} is of the same order of magnitude as the bond energy of the corresponding anions with H^+.

Present evidence, while inconclusive, points to retardation of electron transfer between complexes by hydration-sphere ligands. Prasad and Bavdekar (*33*) found that the quantum yield for the photoreduction of Fe^{+3} to Fe^{+2} for solutions of $FeCl_3$ was twice as great in anhydrous ether as it was in aqueous acetone, again indicating the "protective" nature of the solvation sheath. Bezier (*34*) found that the concentration of ferrous ion was without effect on the electrode potential of dilute solutions of Fe^{+3} where the anion was nitrate or perchlorate; *i. e.*, noncomplexing. With anions that substitute in the hydration sphere, chloride and sulfate, the potential was found to be lowered. It is hoped that continued exploitation of radioisotopy and polarography will shed additional light on the problems of electron-transfer mechanisms and electron transitions in ionic complex configurations, for it is precisely these functions that hold the key to such diverse problems as inorganic color and reaction mechanism.

COLOR AND STRUCTURE

The problem of the relation of inorganic color to structure is an intriguing one. Although the relation of constitution to color for organic compounds has been established on a coherent, semiempirical basis which has proved sound enough to enable fairly accurate prediction of the color of projected compounds, the comparable state of color theory for the inorganic complexes and organo-metallic salts is fragmentary and unsystematized (*35*, *36*). In recent years, as data on inorganic colored complexes have accumulated, it has become increasingly clear that color cannot be correlated with structure by a single general theory (*37*); apparently there are manifold structural causes giving rise to color. However, as Wells (*38*) has indicated, it should be possible to establish "class" correlations as a function of specific bond types. Whether such phe-

nomena as the color-fading of intensely colored complexes like ferric thiocyanate with increasing acidity are due to the formation of an aquo-acid, anionic substitution, or other restrictions in electron environment appears to be a fertile field of investigation.

The relation of color changes to structural changes has already been pointed out, and one of the aspects of this problem that has received recent attention has been the intense coloration due to the existence of a metallic ion of apparently intermediate valency. Thus, the existence of two substances in chalcopyrite: $Cu^{++}Fe^{++}S_2$ and $Cu^{+}Fe^{+++}S_2$, originally assumed by Pauling and Brockway (*39*) as the color-generating equilibrium has since been confirmed (*40*). The role of this type of transition in the structural problems of the ferri- and ferrocyanides has been recently reviewed by Callis (*41*). This latter field of study also provides us with the link between the colloidal solution and the true solution. The mechanism of transition is apparently one of supercomplex formation or polymerization. In fact, even in solutions of a "simple" salt, supercomplex formation may contribute to the coloration; von Kiss (*17*) detected polymerization of the $Fe(SCN)_6^{-3}$ complex in concentrated solutions of the ferric salt.

This polymerization may quite possibly be another manifestation of the phenomenon of resonance stabilization. Recent evidence indicates that intermetallic compounds, with their "odd" valences, are stabilized through this mechanism. The intense color of the solutions discussed above, together with their tendencies to "grow" in solution, are indications of a resonance structure analogous to the intermetallic compounds. However, no definite statement as to structural mechanisms may be made until further research resolves the problems of solution structure.

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THE HARCOURT BUTLER TECHNOLOGICAL INSTITUTE

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Scientific and technological research has been utilized to the advantage of industry principally in the Western countries. Industrial development in India is at a very low level, mainly because science is studied in the university laboratories only and very little attempt has been made to bridge the gulf between science and industry. This has necessitated the establishment of a large number of technical and research institutes and the development of the older institutes along modern lines.

HISTORY

The need for such institutions in this country was recognized as early as 1907, when the Industrial Conference recommended to the Government of the United Provinces the establishment of a technological institute. This recommendation, though accepted by the Provincial Government as well as by the then Secretary of State for India, could not be carried out, for various reasons, until 1920, when under the able guidance of Dr. E. R. Watson a research institute was established at Kanpur. In 1921, on the recommendation of the Indian Industrial (Holland) Commission, the training of students was started at the Institute, which then assumed the name of Harcourt Butler Technological Institute, after the Governor of the United Provinces.

In the beginning a three-year Post Graduate Diploma Course was started in two sections: (1) Chemical Research, and (2) Oil Chemistry and Technology. The first-year course was common to both the sections while the second and third years were devoted to specialization in individual subjects. In 1922–23 a section of Leather Chemistry and Technology was added. In 1926 another section of Sugar Technology was added on the recommendation of the Economic Committee of 1923. In 1929 the admission qualification was lowered to the I.Sc. and training was given for three years. However, the disadvantage of this was soon realized, and in 1932, on the recommendation of the enquiry committee appointed by the Government, the minimum qualification for admission was again raised to the B.Sc., but the period for acquiring the diploma of A.H.B.T.I. (Associate of the Institute) was reduced to two years. Another two-year research course was also started, to follow the Associate diploma and to lead to the Fellowship (F.H.B.T.I.). In 1933 the Leather Section was abolished, but short-term courses in the Sugar and Oil Sections were started for the benefit of those not intending to cover the whole two-year course. In 1936 the Sugar Section was taken over by the Indian Council of Agricultural Research and made into a full-fledged Institute itself.

Recently research chemists for vegetable fibers, essential oils, and plastics have been appointed to the General Research Section. The Government of India has also decided to award technical scholarships to the Institute for further training in India. The U. P. Scientific and Research Committee has awarded 11 scholarships for industrial research work. The Institute also makes several scholarships and fellowships available to its own students.

ORGANIZATION AND FUNCTIONS

The Institute is run by the Department of Industries of the Government of the United Provinces. The Provincial Government administers the Institute through the Director of Cottage Industries, U. P., on the recommendations of an Advisory Committee, representing various Governmental departments, industries, etc.

The activities of the Institute are broadly outlined as follows:

The Harcourt Butler Technological Institute, Kanpur, India

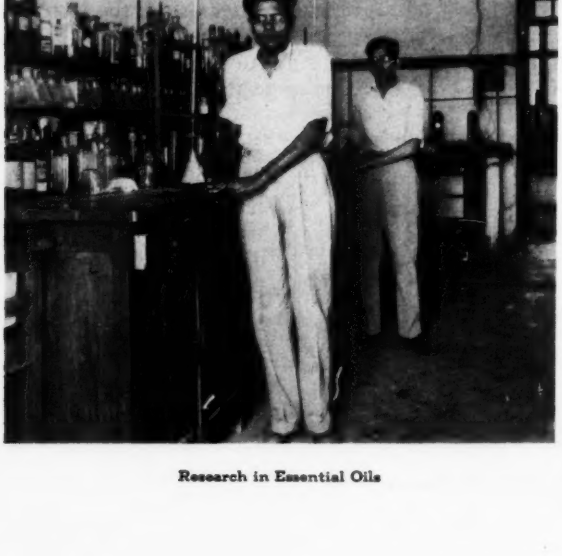
1. Training of students for the diplomas of A.H.B.T.I. and F. H.B.T.I. and short-term courses.
2. Investigation of raw materials for industries.
3. Standardization of raw materials, processes, and finished products.
4. Exploration of the practical application of the new results of research with a view to their use in industry.
5. Testing of industrial and excise samples of commercial importance.
6. Technical advise to various industries in the Province.
7. Publication of technical bulletins and scientific research papers.

COURSES OF STUDY

Associateship Diploma Course (*A.H.B.T.I.*). The course is of two years' duration and the minimum admission qualification is a B.Sc. degree. This diploma has been recognized by the Government as equivalent to an M.Sc. for recruitment by Provincial and Federal Public Service Commissions.

In the General Research Section the first-year students are trained in applied biological and physical chemistry, chemical engineering, and applied practical work. They are promoted to the second year on the basis of a written examination. During the second year they carry on research work on problems of industrial chemistry and at the end they are awarded the diploma of Associateship in Chemical Technology on the result of a thesis.

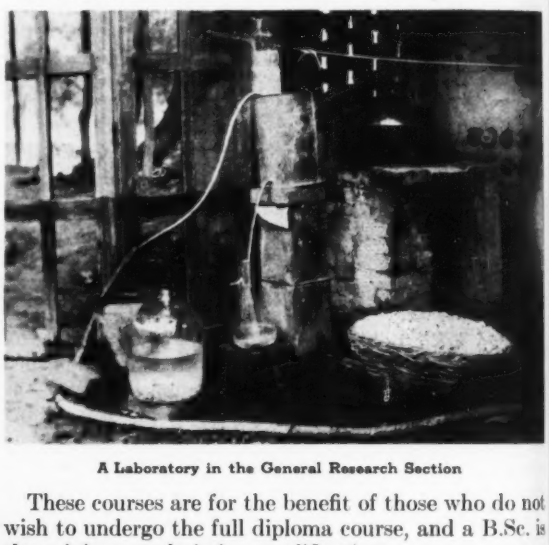
In the Oil Section the course covers a number of subjects, such as oil milling; refining and hydrogenation of oils; soap; paints and varnishes; applied biological and physical chemistry; mechanical, electrical, and chemical engineering, including machine drawing; examination and analysis of raw materials and finished products; etc. The Associateship Diploma in Oil Chemistry and Technology is awarded on the results of a written examination.



Research in Essential Oils

Fellowship Diploma Course (*F.H.B.T.I.*). This is open to A.H.B.T.I. students only. The students in the General Research Section carry out higher research work in chemistry on problems of industrial importance, while in the Oil Section they do higher research work on problems connected with oil and allied industries. At the end of two years the diploma of Fellowship is awarded on the result of a thesis.

Short Courses. There are three short courses of six to eight months each in the Oil Section. They are Oil Milling, Soap Manufacture, and Paints and Varnishes. There is also a short-term course in Essential Oils and Synthetic Aromatics of six to eight months duration in the General Research Section. Recently the Department has recommended to the Government the addition of two more short-term courses in the latter Section: (1) Industrial Analysis and Testing, and (2) Preparation of Industrial Products.



A Laboratory in the General Research Section

These courses are for the benefit of those who do not wish to undergo the full diploma course, and a B.Sc. is the minimum admission qualification.

RESEARCH PROJECTS

In addition to the teaching work, the following research projects are under way in the General Research Section.

Fiber Project. The work on vegetable fibers was started in 1924, but with the division of the country the need of a proper substitute for jute fiber (mainly grown in Pakistan) has become so urgent that a full-time research chemist is now engaged in this project. The results achieved so far with linseed, banana, aloe, bhang, and calotropis are very encouraging; so much so that shortly attempts will be made to exploit linseed fiber on a commercial scale as a substitute for jute.

Essential Oils. Considerable work has been done in reviving the old perfume industry of this Province by developing a new method of making attars from flowers of kewda, rose, bela, chameli, harsingar, mahdi, and maulsari. These attars have secured new markets in foreign countries.

Plastics. This project has been going on for the last two years at the Institute with a full-time research chemist employed. Plans for the extensive cultivation of lac have developed, with the cooperation of the Indian Lac Research Institute at Ranchi. Several new molding powders from molasses and proteins have been prepared to replace cheaply and locally the foreign powders which the mills were hitherto using and which are difficult and costly to obtain. Research has also been started on the development of ion exchangers from indigenous tannin materials obtained from our forests.

Handmade Paper. Special efforts have been made to help in the development of the handmade paper industry by investigating the possibility of using various cellulosic materials. Kans fiber has proved very useful for the manufacture of paper and boards.

Cottage Oil. A project for the development of the cottage oil industry is being carried out in the Oil Section. Some improvements in Wardha Oil Kolhu have been made at this Institute. Necessary literature and drawings are supplied to popularize the use of improved wardha oil ghani instead of old indigenous ghani at present used by village telis.

DETERMINATION OF THE RANGES AND CONSTANTS OF DOUBLE-RANGE INDICATORS

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University College of Khartoum, Sudan

MOST textbooks on practical physical chemistry include an experiment on the determination of the constant pK_{In} and range of a single-range indicator (*1*). Finding that these very instructive exercises aroused considerable interest in the students we followed up experiments on phenolphthalein, a single-colored indicator, and methyl red, a two-colored indicator, by an unknown indicator having 2 ranges and 3 colors.

The indicators (*2*) and buffer solutions[1] used were:

Thymol Blue

pH range:	1.2–2.8	8.0–9.6
Color change:	Red–yellow	Yellow–blue
pK_{In}:	1.7	8.9

Buffers: 1.1, 1.4, 1.7, 2.0, 2.5, 2.7, 3.1, 3.6, 7.0, 7.5, 7.8, 8.0, 8.3, 8.6, 8.8, 9.1, 9.5, 9.8

o-Cresol red

pH range:	0.2–1.8	7.0–8.8
Color change:	Red–yellow	Yellow–purple[2]
pK_{In}:	1.0	8.3

Buffers: 0.0, 0.3, 0.6, 0.9, 1.2, 1.4, 1.9, 2.3, 6.5, 6.8, 7.1, 7.3, 7.6, 8.0, 8.2, 8.3, 8.8, 9.1

m-Cresol Purple

pH range:	1.2–2.8	7.4–9.0
Color change:	Red–yellow	Yellow–purple
pK_{In}:	1.5	8.3

Buffers: 1.0, 1.2, 1.4, 1.6, 1.9, 2.3, 2.5, 2.7, 2.8, 3.1, 3.7, 7.5, 7.8, 8.0, 8.3, 8.5, 9.0, 9.5

Stock solutions of the indicators were prepared by triturating 1 g. with 0.1 *N* NaOH and diluting with water to 1 liter. The volumes of NaOH taken were: thymol blue, 21.5 ml; cresol red, 26.2 ml; cresol purple, 26.2 ml.

The instructions given were:

Take two racks with 18 test tubes and add one drop of indicator to each. Prepare, and add to each tube in turn, 10 ml. of the buffer solutions of the series given. Note the two ranges of pH and the colors. Place the two tubes possessing the extreme colors of the first range behind each other and look through them—the intermediate color of this range will be seen. Compare with the other tubes. The tube which matches this color gives the indicator constant, *i. e.*, its pH value = pK_{In}; explain why. Repeat for the second range. Look up a list of indicators (*3*) and decide which indicator you have been given.

Most textbooks give five buffers as being sufficient to obtain the range and constant of one indicator or one range, but we find that nine take very little extra time to prepare and the results obtained are greatly improved. The average results were extremely good, being no more in error than ± 0.2 pH unit. One drop of the indicator solutions quoted is sufficient for the 10-ml. quantities of buffers; burets are very useful for delivering drops of constant volume.

[1] pH 0–1: Known HCl solutions used being quite good enough for this purpose where little buffering power is required. pH 1–2: HCl + di-sodium hydrogen citrate, (ref. (*1*), p. 232). pH 2–10: Addition of HCl or NaOH to "British Drug Houses" Universal Buffer (*4*).

[2] The alkaline color of *o*-cresol is always given as red but we find it quite definitely purple having exactly the same shade as the alkaline color of *m*-cresol purple.

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CASTOR OIL AS A STARTING MATERIAL FOR LABORATORY PREPARATIONS

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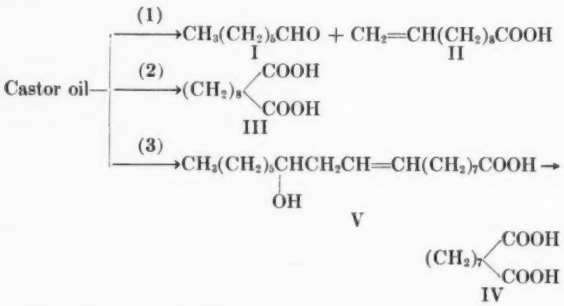
Technological Institute for Advanced Studies, Monterrey, Mexico

Castor oil is a readily available natural substance which, by thermal degradation or oxidation, gives four derivatives, I, II, III, IV, which in turn can easily be converted into a number of interesting compounds, using procedures which are in general reliable, do not involve unusual risks or complicated methods, and give yields ranging from 40 to 60 per cent. These compounds also can be employed in serial reactions in a manner rarely found in the usual preparations of the organic chemistry laboratory.



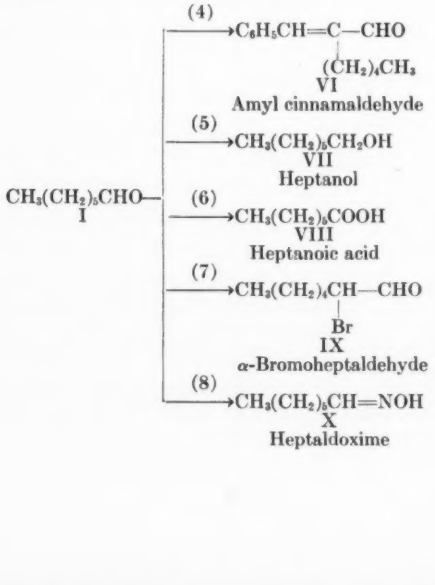
The thermal decomposition of castor oil with the isolation and identification of heptaldehyde (enanthol) was first carried out by Bussy (*1*) in 1846. Five years later Bouis (*2*) added potassium hydroxide prior to the distillation and obtained sebacic acid. Stanek (*3*) and Leeds (*4*) prepared several derivatives of heptaldehyde. In 1877 Krafft (*5*) identified the second component of the decomposition as undecylenic acid and seven years later found that the amorphous residual paste of the distillation is a polymer of undecylenic acid which, by oxidation with nitric acid, gives sebacic acid. A modification of the thermal decomposition of castor oil has been employed by Grun and Wirth (*6*) who started with the crude esters of recinoleic acid and obtained the ester of undecylenic acid with a 68 per cent yield.

Reaction 1. In a Claisen flask are mixed 150 g. of castor oil and 10 g. of colophony; the usual connections for vacuum distillation are set up and the flask is heated with a bare flame. After 6 to 10 minutes the distillate begins to collect in the receiving flask. When approximately 60 to 80 ml. have been collected, the heating is stopped (to avoid the formation of the yellow resinous paste in the flask, which is very difficult to clean). If two layers are formed in the distillate, the lower layer (water) is taken out in a separatory funnel and the upper layer is poured into a 500-ml. flask to which is attached an efficient rectification column; the distillate passing between 145° and 160°C. is collected (crude heptaldehyde) and, when the temperature rises above 170°, the distillation is interrupted. The crude heptaldehyde (I) is purified by a second distillation (not necessary if it is going to be used as a starting material for subsequent reactions). The yield is about 24 to 30 g. The residue of the fractional distillation is distilled in a vacuum. The undecylenic acid (II) distils between 156 and 165° at 30 mm., giving a yield of 25 to 30 g.

Reaction 2. Castor oil is heated at 40° for one hour with a 40 per cent solution of sodium or potassium hydroxide. When cooled, the upper layer is separated, mixed with solid sodium (or potassium) hydroxide, and heated in a copper or iron basin until the odor of octano-2 vanishes (three hours). It is then poured into cool water and the solution is acidified with hydrochloric acid. Sebacic acid (III) is collected by suction (*7*), the yield being 20 to 40 per cent.

Reaction 3. Castor oil is saponified with a methanolic solution of potassium hydroxide, and the recinoleic acid (V) formed is immediately oxidized with an alkaline solution of potassium permanganate. After acidification and purification a yield of 32 to 36 per cent of azelaic acid (IV) is obtained (*8*).

Heptaldehyde (I) undergoes a large variety of reactions, of which the following are characteristic:



Reaction 4. Condensation of benzaldehyde with heptaldehyde in a solution of sodium hydroxide. The yield of amyl cinnamaldehyde obtained is 60 to 70 per cent (*9*).

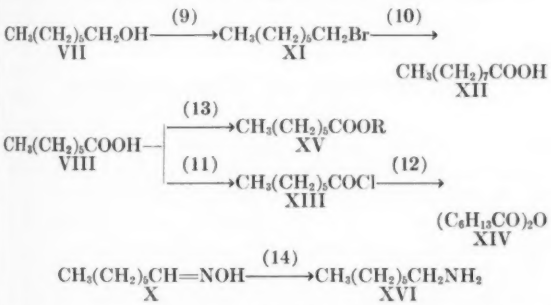
Reaction 5. Reduction of I with iron and acetic acid. Heptanol is formed; yield, 75 to 80 per cent (*10*).

Reaction 6. Heptaldehyde is oxidized with potassium permanganate in an alkaline medium, and product VIII is isolated by acidification. It can also be prepared by refluxing I with a solution of sodium dichromate in sulfuric acid. In both cases the yield is 65 to 75 per cent (*11*).

Reaction 7. Heptaldehyde is converted with acetic anhydride and potassium acetate to the heptaldehyde enol acetate (yield, 45 to 50 per cent), which is brominated and treated with methanol to give α-bromoheptaldehyde dimethyl acetal (yield, 80 to 85 per cent), which, with diluted hydrochloric acid, is converted to IX; yield, 90 per cent (*12*). These reactions should only be carried out by advanced students, because they involve the preparation of lachrymous compounds, and require careful control.

Reaction 8. Compound I is condensed in an alcoholic medium with hydroxylamine hydrochloride in the presence of sodium carbonate, and heptaldoxime (X) is obtained; yield, 80 to 90 per cent (*13*).

Compounds VII, VIII, and X are readily converted to a number of interesting compounds, all of which are sufficiently reactive to be used in secondary syntheses. Some of the more interesting reactions are as follows:



Reaction 9. Compound VII can be refluxed with a mixture of red phosphorus and bromine (*14*) or with anhydrous bromhydric acid (*15*), or with a sulfuric acid-sodium bromide mixture, giving a 75 to 90 per cent yield of heptyl bromide (XI).

Reaction 10. Through an intermediate malonic condensation XI is converted to pelargonic acid (XII). The over-all yield is 60 to 75 per cent (*16*).

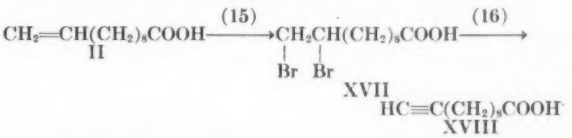
Reaction 11. Compound VIII is refluxed with phosphorus pentachloride. Phosphorus oxychloride distils over at 107° and heptoyl chloride (XIII) at 170–180°, according to Lumsden (*17*). Alternatively, VIII may be refluxed with thionyl chloride, the heptoyl chloride formed being purified by fractional distillation; yield, 70 to 85 per cent.

Reaction 12. Pyridine is added to a benzene solution of VIII and XIII, and, after refluxing and purification, heptoic anhydride (XIV) is obtained; yield, 70 to 80 per cent (*18*).

Reaction 13. A solution of VIII in an excess of methanol (or ethanol) containing 10 per cent of sulfuric acid is refluxed for three hours, then poured in water. The upper layer is washed several times and purified through fractional distillation, giving a 75 to 85 per cent yield of XV.

Reaction 14. An alcoholic solution of X is reduced with sodium, giving, after purification, a 50 to 70 per cent yield of heptyl amine (XVI) (*19*).

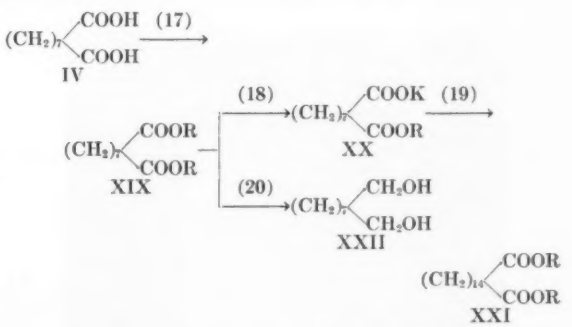
Undecylenic acid (II) may react as follows:



Reaction 15. Bromine is added to a petroleum ether or carbon tetrachloride solution of II. The dibromide (XVII) precipitates and can be collected with a yield of 85 to 95 per cent (*20, 21*).

Reaction 16. A suspension of XVII in glycerol-potassium hydroxide solution is heated for two hours at 100°, then the solution is diluted with water and acidified, when dehydro-undecylenic acid (XVIII) is formed. When purified, the yield is 60 to 75 per cent (*20, 21*).

Using III or IV as starting materials homologous compounds can be obtained. The following illustrates the use of IV, the products obtained from III being similar.



Reaction 17. Ten per cent of concentrated sulfuric acid is added to a solution of IV in methanol (or ethanol). The mixture is refluxed three hours, poured in water, and the organic layer is washed, dried, and vacuum distilled. The first fraction is the diester, the second is the monoester. The yield of the diester (XIX) ranges between 50 and 70 per cent (*22*).

Reaction 18. An alcoholic solution of XIX is allowed to stand for 24 hours after the addition of the theoretical amount of a 2 *N* alcoholic potassium hydroxide solution for the saponification of one of the ester groups. Compound XX is obtained with a yield of 30 to 45 per cent (*6, 23*). Another and better pro-

cedure is the acidolysis of the diester using anhydrous hydrochloric acid as catalyst (*24*); yield, 45 to 65 per cent.

Reaction 19. By electrolysis of XX in an alcoholic solution at 50–60°, using platinum electrodes and a current of 3 amperes and 30 to 60 volts, hexanodioic acid (XXI) is obtained; yield, 30 to 40 per cent (*23, 25*).

Reaction 20. The diester XIX is reduced in alcoholic solution with sodium, and the glycol is extracted with ether and purified by vacuum distillation, to give a 50 to 60 per cent yield of XXII. As an alternative to the reduction lithium aluminum hydride can be employed (*22*).

The products obtained in all these reactions can be converted to other interesting derivatives, both aliphatic and aromatic.

ACKNOWLEDGMENT

The authors wish to express their appreciation for the cooperation and advice of Ing. Carlos Duhne and Ing. Adalberto Reiter, and to the students Berta Gutierrez and Erika Homberg for their help in the experimental work.

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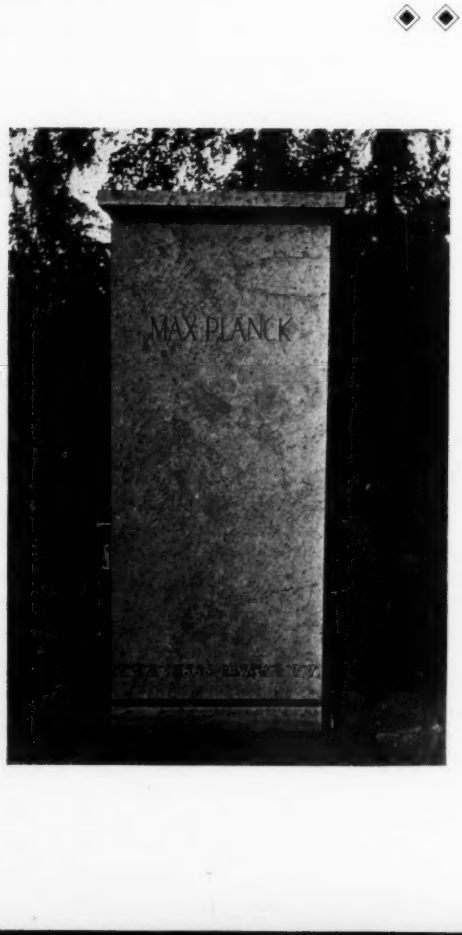

GRAVESTONE OF MAX PLANCK

Max Planck (b. April 23, 1858, Kiel) lived and worked in Berlin from 1889. In World War II his house went up in flames during an air raid, and his library, collected throughout his exceptionally long lifetime, disappeared, no one knows where. In the middle of May, 1945, the American authorities sent a car to his estate of Rogätz on the Elbe, then a theater of war, and took him to Göttingen, where he would be safe. Here he died on October 4, 1947, almost four-score-and-ten.

The mortal remains of this great theoretical physicist, the author of the quantum theory, rest in the Göttingen Stadtfriedhof. The grave is fittingly marked by a simple stone, bearing nothing more than his name, and at the foot the Planck constant:

$$h = 6.62 \cdot 10^{-34} W \cdot s^2$$

($W \cdot s^2$ = watt second^2)

—*Ralph E. Oesper, University of Cincinnati, Cincinnati, Ohio*

THE HIGH POINTS IN THE DEVELOPMENT OF CELLULOSE CHEMISTRY SINCE 1876[1]

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La Jolla, California

WHEN cellulose chemistry entered the founding year of the A. C. S. in 1876, an astonishing wealth of essential facts were then known. One of the most consequential events would appear to be the discovery by the French chemist Braconnot in 1819, namely, that wood and purified linen yield glucose under the action of strong as well as dilute sulfuric acids. It was through this discovery that the foundation was laid for all further studies concerning the nature of cellulose and its molecular structure.

Of similar importance were the labors of another Frenchman, Anselme Payen, eighteen years later, in 1837. Payen recognized the fibrous portion of plant tissue as its "skeleton" substance. When he gave it the name "cellulose" (which he derived from the Latin word "cellula" for small cell), he wished to express the view that the fibrous substance represented a uniform chemical individual and was not a constituent of any of the other components of the plant, for example, the lignin in wood or the pectin in cotton, and the like. Payen had to fight long and hard to defend his then rather progressive and fertile views. However, in the end, he saw them accepted by the majority of his contemporaries.

About 40 years later, in 1880, Cross and Bevan, the two pioneering English cellulose chemists, to whom we otherwise owe so much in the cellulose field, undid Payen's accomplishments by postulating and getting accepted their belief that the entire fiber rather than the cellulose alone represented a chemical individual. Later on, they held that cellulose forms different compounds with the noncellulosic substances, such as lignin or pectin, depending upon the plant material under investigation. Cross and Bevan thus distinguished between various types of cellulose, as *e. g.*, "lignocellulose" and "pectocellulose." It took many years before the cellulose chemists gave up this idea and returned to Payen's original concept, *i. e.*, that cellulose existed by itself as an individual. This view of the nature of cellulose does, of course, not exclude the possibility that in the original plant material cellulose is chemically combined with lignin or with hemicelluloses. However, today we look at these substances rather as contaminations, *i. e.*, substances which can be removed without essentially changing the nature of cellulose.

[1] Address at the Banquet of the Division of Cellulose Chemistry at the 75th Anniversary Meeting of the American Chemical Society, New York, September, 1951.

Another important fact prior to 1876 was Braconnot's conversion in 1833 of cellulose by the use of anhydrous nitric acid into nitrocellulose, an inflammable derivative, from which also tough and glass-clear films could be produced. A few years later, in 1844, we register John Mercer's discovery of the changes which occur to cellulose under the action of strong aqueous alkali and which after him were collectively named "mercerization." Mercer also discovered that cellulose, which otherwise was found to be insoluble, may be dissolved in an aqueous solution of cuprammonium hydroxide. About thirteen years later, in 1857, Edward Schweizer made the same discovery, without knowing Mercer's work, but went more deeply into the matter and established a sound basis for further studies. In 1847 Schönbein laid the ground for the commercial process of cellulose nitration by introducing concentrated sulfuric acid as a catalyst.

Of similar far-reaching consequence was Schützenberger's use of acetic anhydride for the preparation of cellulose acetates in 1869 and the fact that he recognized the number three as the highest degree of substitution for each glucose residue in cellulose. This had been even earlier recognized for the conversion of cellulose into cellulose nitrate, by W. Crum, in 1847. During the years which followed, the concept of the existence in each glucose residue of only three available hydroxyl groups (which may be converted into esters, ethers, and other derivatives) fell into oblivion and was rediscovered only very much later.

The year 1847 brought to light another important fact. It was then that J. H. Gladstone, probably as the first, suggested that the process of nitration represents an equilibrium reaction. However, no attention was paid to Gladstone's interpretation, until many years later when it was again suggested by Ernst Berl and subsequently helped much to elucidate the mechanism of nitration.

HYDRO AND OXYCELLULOSE

The various facts which I have briefly sketched represent the general standing of cellulose chemistry when the American Chemical Society was founded in 1876.

This, its first year, was quite appropriately inaugurated by the introduction through the French chemist Girard of the term "hydrocellulose" for cellulose which had suffered loss in physical strength under the action

of dilute aqueous mineral acids. Girard recognized this modification of cellulose as the first intermediate on its conversion under the action of such acids into glucose. That this modification contained no water in chemical combination with the cellulose, as Girard had believed and for which reason he thought "hydrocellulose" to be an appropriate term, was clarified only many years later, namely by Stern in 1904 and by Ost in 1913.

It is interesting to note that the question as to whether hydrocellulose possesses higher reducing power than the original fiber was disputed for quite some time, until C. G. Schwalbe eliminated any doubt through his quantitative investigations of 1907, through which he also introduced the term "copper number" and a technique by which to obtain reproducible results.

The early years of the Society also witnessed the discovery and coining of the term "oxycellulose" by Witz in 1883 for another commercially undesirable modification of cellulose, observed in fabrics and other processed celluloses that had become tendered during bleaching, as a result of oxidation. It is reported that Cross and Bevan, who visited Witz at the time of the latter's discovery, participated in the coining of the term.

Witz's studies initiated the now historical investigations on the nature of oxycellulose by Nastukoff, Vignon, Tollens, and Berl between 1883 and 1907, with their opposing views, namely, whether oxycellulose was a uniform individual or a nonuniform mixture. Thus, for Tollens and Vignon, whose views predominated for most of those years, oxycellulose was an individual compound in which a varying number of unchanged $C_6H_{10}O_5$ units was in ether-like combination with its oxidized component, called "celloxin" by Tollens, and assumed to represent a glucoronic acid lactone (glucorone, $C_6H_{10}O_6$). This view was in agreement with the observation that oxycellulose, on boiling with dilute hydrochloric acid, liberated furfural and carbon dioxide, a reaction which was first observed by Cross, Bevan, and Beadle in 1893. Although Berl and Klaye's study in 1907 made the interpretation of oxycellulose as a chemical individual rather improbable, it prevailed until the early 20's of our century, when F. Stöckigt in the Institute of Cellulose Chemistry in Darmstadt, in 1922, and Hibbert and Parsons at McGill University in Montreal, in 1925, produced enough experimental evidence to show that the usual oxycellulose preparations, known at that time, were heterogenous mixtures of unchanged cellulose with low-molecular-weight oxidation products. Further clarification of the oxycellulose concept resulted from the classical studies of the British textile chemists at the Sherley Institute in Manchester, such as Clibbens, Ridge, Geake, Birthwell, Neale, and, most prominently, G. H. Davidson, on the effect of the nature of the oxidant and the hydrogen ion concentration and from the distinction they made between the reducing and the nonreducing types of oxycellulose. The most important of these studies were made between 1923 and the late 1930's.

A new era of oxycellulose chemistry, which has produced new types of oxycellulose and thrown much light upon the mechanisms involved, dates from 1937, when C. S. Hudson produced the first oxycellulose which may be looked upon as a chemical individual. Hudson and co-workers found that under the action of periodic acid, the hydroxyl groups on the carbon atoms in the 2- and 3-positions are oxidized to a dialdehyde grouping. Another individual oxycellulose was produced by Kenyon and his co-workers at the Eastman Kodak Company in 1942 by subjecting cellulose to oxidation with nitrogen dioxide, whereby the hydroxyl groups in the 6-positions are oxidized to carboxyl groups. During the years which followed these discoveries, Milton Harris, Rutherford, Purves, Mark, Staudinger, and their numerous co-workers participated in the elucidation of the mechanisms involved in the preparation and in the reactions of these new oxycellulose types.

TOLLENS' FORMULA

Another important discovery which falls into the early years of the Society was the introduction, by Franchimont, of sulfuric acid as a catalyst for the acetylation of cellulose with acetic anhydride in 1879. The crystalline acetylated by-product which Franchimont isolated from the reaction mixture and believed to be an 11-fold acetylated trisaccharide was later recognized as the octa-acetate of cellobiose by König and Skraup in 1901. The importance which the isolation of this first crystalline and well-defined intermediate of cellulose degradation had for the molecular structure of cellulose was well recognized by Skraup and co-workers, by Franchimont in his later studies of 1899, and by Ost and co-workers during the latters' investigations between 1901 and 1907. The discovery of cellobiose was regarded as a firm support of the formula which Tollens had published some years earlier, in 1895. This formula showed the anhydroglucose units to be united by double oxygen (acetal) linkages in such a way as to form a chain molecule in which three hydroxyl groups were available for successive esterification and etherification. It is interesting to note that Tollens had suggested the chain formula idea for cellulose as early as 1880 and that he had also conceived of a successive cleavage of the acetal linkages in cellulose on hydrolysis until the entire molecule was converted into glucose. Thus Tollens' formula not only explained the formation of glucose and cellobiose but also showed that other intermediates could be expected on the degradation of cellulose to glucose.

The immediate tasks which the Tollens formula thus called for were the determination of the molecular structure of cellobiose, the isolation of the intermediates with more than two anhydroglucose units, and the elucidation of the structure of such higher intermediates.

However, before these tasks were undertaken, cellulose chemists—apart from establishing definite proof for the quantitative conversion of cellulose into glu-

cose—devoted their interest mainly to two other items. One of these was Curt Hess's hypothesis of 1920 which abandoned the concepts of classical organic chemistry and, instead, proposed that the glucose residues are held together by secondary valence forces. Although the excitement over this hypothesis, the polemics which followed, and the eventual victory of its opponents—the "rise and fall of the association theory," as Purves has put it—meant much delay for the normal development, this period produced a wealth of experimental data; in fact it gave cellulose research a new impetus and inaugurated a new era of activity.

MOLECULAR WEIGHT

The other item of interest was the viscosity of cellulose in solution and its relation to the degree of cellulose degradation, particle size, or molecular weight. It is likely that Wilhelm Ostwald in 1909 was the first to postulate that internal friction of solution of a colloid such as cellulose—that is its viscosity—increases with increasing molecular weight and vice versa. At about the same time Ernst Berl and co-workers established the existence of such a relationship for cellulose dissolved in cuprammonium hydroxide and for cellulose nitrate dissolved in acetone. In 1910 Berl and Bütler reported that they had found the viscosity of a 5 per cent nitrocellulose solution to be 9000 times higher than that of a starch nitrate dissolved at the same concentration. From this, Berl and Bütler concluded that cellulose very likely possesses a much higher molecular weight than starch, a prediction which was soon verified. In 1911 Ost, and in 1920 W. H. Gibson, placed the relationship between molecular weight and viscosity on a more quantitative basis. In 1925 Staudinger entered the stage by advancing the postulate that there exists an absolute relationship between viscosity, molecular weight and degree of polymerization (D.P.), i. e., the number of anhydroglucose units per cellulose chain.

The rapid development that followed is well recalled. It is characterized (1) by a period of criticism and polemics, which, however, contributed much to the perfection of the techniques to be used and to a clarification of concepts; (2) by the proposition of the late Elmer Kraemer in 1938 to base the K_m constant in the Staudinger function on ultracentrifugal (instead of osmotic pressure) data; and (3) the eventual acceptance of the Staudinger function and its modification as useful and practical tools for the characterization of cellulosic materials within certain limits of degrees of polymerization. At about the same time, the concepts of "average chain length" and of "chain-length distribution" or "polymolecularity," which were derived from the results of the fractionation of cellulose, mostly in the form of its derivatives, were developed. These concepts referred to the presence in a given cellulose sample of medium, long, and short chains.

Likewise, other methods for the determination of the molecular weight of cellulose were brought to a greater perfection; thus, the osmotic pressure method, particularly by the studies of Duclaux and Dobry during the period from 1911 to 1929, and the ultracentrifugal techniques by Svedberg of 1927. These latter techniques were first applied to cellulose by Stamm in 1930, put on a more reliable basis by Kraemer and Lansing in 1934, and further developed by Gralén and by Jullander in recent years. The latest method of expressing viscosity-molecular-weight relationships is based upon the scattering of light. It was suggested by Debye and adapted for cellulose in suspensions and solutions by Doty and Mark five to six years ago.

Before going further in depicting the highlights of cellulose chemistry during the last 57 years, I should like to record the appearance in 1911 of C. G. Schwalbe's book, "The Chemistry of Cellulose," as a high point of the decade from 1910 to 1920. Whereas the volumes known as the "Cross and Bevan Researches," which were then the only source of cellulose chemistry in book form, consisted merely of periodical reviews of selected subjects, Schwalbe's book was the first complete compilation of what was known of cellulose chemistry at that time. In it were also registered, among other interesting items, the then newest "highlights," such as Cross and Bevan's discovery, in 1892, of the cellulose xanthate and of the manufacture of viscose, as well as the preparation of acetone-soluble acetates by the Miles process in 1904. Undoubtedly, Schwalbe's book has acted as a great stimulus for the research activities of the years which followed its appearance.

MOLECULAR STRUCTURE

The year 1926 brought the fulfillment of the two most essential prerequisites for the transformation of the Tollens formula into the concept now generally accepted. These were first, the clarification of the molecular structure of cellobiose as a 1,5-glucosido-1,4-glucose by Haworth and his school, and second, the isolation from the acetolyzate of cellulose of the first oligosaccharide, cellotriose, in crystalline form by Ost, after this trisaccharide, in its amorphous form, had been discovered by Bertrand and Benoist in 1923. The isolation of other oligosaccharides followed: cellotetraose by Willstätter and Zechmeister in 1929, and cellohexaose by Zechmeister and Tóth in 1931. The most recent work concerning the oligosaccharides of cellulose comes from Wolfrom and Dickey, who in 1949 succeeded in isolating from the acetolyzate of cellulose a complete polyhomologous series of the sugar acetates—that is, from the acetate of cellohexaose to that of glucose—by their chromatographic technique. They have recently added one more member to the series, celloheptaose.

The studies of Haworth and of Freudenberg and their co-workers during the 1930's established the molecular structure of the oligosaccharides; they showed that in all of these intermediates the anhydroglucose units are linked by 1,4-glycosidic bonds, as in cellobiose.

At this juncture it would seem to be appropriate to

recall that the clarification of the molecular structure of cellobiose and the oligosaccharides was made possible, or at least much facilitated, by using, for the hydrolytic and acetolytic studies, cellulose in the form of its methyl ether. The latter could thus be converted into the fully or partially methylated sugars, in which the position of the methyl groups could be easily determined. Much successful pioneering in this field was done in England by Irvine, Hirst, and other members of the Irvine school in the early 1920's and in Germany by Curt Hess and co-workers at about the same time. Although methylated cellulose appeared first in a study by Suida on surface-methylated cotton in 1905, the first fundamental investigation was made by Denham and Woodhouse in 1913, followed by further studies of these authors in 1914 and 1921. We also recall that it was methylation with subsequent hydrolysis which led to the discovery by Haworth and Machemer in 1932 of the long sought for tetramethyl glucose, a discovery which demonstrated that the cellulose molecules may be looked at as open chains.

The last of the prerequisites for a satisfactory molecular structure of cellulose, namely a clarification of the prevailing configuration, was fulfilled first by Freudenberg's synthesis, in 1923, of cellobiose and cellotriose derivatives from compounds in which the configurations were known. This was followed by the results of a study of the kinetics of cellulose hydrolysis by C. H. Meyer, Hopff, and Mark in 1929, by W. Kuhn in 1930, and by Freudenberg, *et. al.*, in the 1930's, and finally by a study of optical rotation and superposition by Freudenberg in 1932. All these results favored the β-configuration as opposed to the α-configuration, as it exists in starch.

Thus, the cellulose formula of today represents a chain of anhydroglucose units, linked together by 1,4-glycosidic linkages of the β-configuration. A more recent question has not been answered yet: namely, whether or not the chain system contains so-called "weak linkages" or "soft spots," other than the 1,4-linkages—as has been proposed by G. V. Schulz.

SUBMICROSCOPIC STUDIES

Before studies of the molecular structure of cellobiose and the oligosaccharides were begun in the early 1920's, cellulose research had received a new impetus from the application of X-ray analysis to the study of the submicroscopic structure of the cellulose fibers. This development, which had started as early as 1913 with the publication of the first X-ray diagram of a cellulose (hemp) fiber by Nashikawa and Ono, but had remained unnoticed, attracted the attention of the European cellulose chemists through the independent studies of A. W. Hull in this country in 1917, but to a greater extent through the X-ray diffraction diagram of a ramie fiber, as obtained and interpreted by Paul Scherer in Germany in 1920. The evaluation of this and subsequent diffraction patterns of other cellulose fibers led to the concept that much of the cellulose may be regarded as "crystalline" and that the cellulose chain molecules are arranged in a crystal lattice in a way similar to the structure of other crystalline substances. However, quite a portion of the cellulose appeared also noncrystalline or amorphous. I may assume that the subsequent highlights of this development are still well remembered, particularly the determination of the dimensions of the unit cell by Sponsler in this country in 1917 and by Polanyi and Herzog in Germany in 1920 and 1921.

As the X-ray work on cellulose went rapidly ahead, it became more and more obvious that its findings had to be brought into line with the also rapidly developing chemistry of cellulose. This goal was accomplished in this country by Sponsler and Dore in 1926 and in Germany by Meyer and Mark in 1928. Meyer and Mark demonstrated that Haworth's then new cellobiose model would fit excellently into Polanyi's dimensions of the unit cell. The arrangement of the cellobiose units opposing each other in the unit cell was slightly changed by Meyer and Misch in 1937.

The evaluation of the X-ray diffraction pattern of the cellulose fiber by the early X-ray workers had led to a revival of von Nägeli's micellar concept of 1857, particularly through the interpretation which Ambronn in 1911 had given the results of his studies on the double refraction of the cellulose fibers. However, the credit for having developed the modern concept of a micellar structure for cellulose is due to Meyer and Mark. This theory found its early expression in Seifriz's well-known bricklike arrangement of 1929, and its later, more satisfactory representation in the models suggested by Frey-Wyssling and by Kratky and Mark in 1936 and 1938. Of the two versions, the Kratky and Mark concept has since been accepted by most cellulose chemists—Staudinger being one of the more notorious exceptions.

As the new concept of the fine structure of cellulose was developed, the interpretation of the microscopic structure of the fiber went through various stages of excitement and controversy until the beginning of the 1940's. At this time, a new era in the elucidation of the invisible structure of cellulose was inaugurated by Ruska through the use of the electron microscope. Although the interpretation of the early results obtained with this new tool likewise had its controversial ups and downs, the basis for a sound evaluation could soon be established. On this basis, the misconceptions which had resulted from a too optimistic view at the object under the light microscope were eliminated.

Another essential contribution to be derived from the use of the new tool would seem to be the excellent support which electron-microscopical observations have given the fibrillar concept of cellulose fibers. The new instrument has been capable of resolving the fibril bundles which may be observed under the light microscope (and which have sometimes been looked upon as ultimate units of the fiber structure) into finer and finer strands. These findings might permit speculation that the ultimate fibril is the single chain molecule.

Whether such speculation can ever be given substance remains to be seen.

For a while it seemed as if the electron-microscopical observations would lead us away from the micellar theory of the fiber structure and favor Staudinger's concept of a simple alignment of chain molecules without fixed dimensions in either direction of the fiber.

However, further progress in electron microscopy in combination with certain chemical and mechanical treatments, such as "limiting hydrolysis" and exposure to ultrasonics, has revealed fibril strands whose lateral and longitudinal dimensions approach those of the hypothetical micelles, as derived from X-ray evaluation. Whether these units are, as Svedberg and Rånby believe to have proved, identical with the hypothetical, X-ray derived micelles is still an unanswered question.

A BRIEF DERIVATION OF THE LEVER LAW

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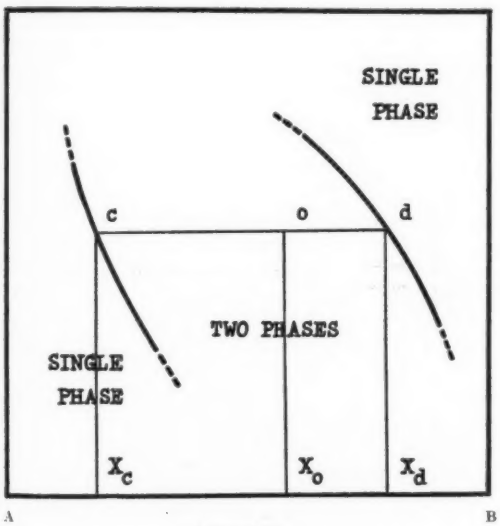
ALTHOUGH the application of the "lever law," or "proportioning rule" as it is also called, is well illustrated in most of the elementary physical chemistry textbooks that I have seen, its derivation is not given. This odd circumstance prompted me to work out a simple and concise derivation.



The accompanying figure represents a typical portion of a temperature-composition or pressure-composition phase diagram of any binary system. If we start with a single-phase system, of composition X_o, and bring it to point o, two phases will result. These are represented by points c and d and have the equilibrium compositions X_c and X_d, respectively. What is the ratio of the amounts of these two phases at equilibrium?

The quantity (either mass or number of moles) of component B in the original mixture is the same as the quantity of B in phase c plus the quantity of B in phase d, since no material transport to or from the system has taken place. That is,

$$Q_{B_o} = Q_{B_c} + Q_{B_d} \tag{1}$$

(Here Q is used to represent quantity, whether mass or number of moles.)

Similarly, the quantity of the entire original mixture is the same as the quantity of the entire phase c plus the quantity of the entire phase d,

$$Q_o = Q_c + Q_d \tag{2}$$

The quantity of B in a mixture of A and B is equal to the quantity of the whole mixture, multiplied by the fraction of the mixture that is B, or

$$Q_{B_o} = Q_o X_o \tag{3}$$
$$Q_{B_c} = Q_c X_c \tag{4}$$
$$Q_{B_d} = Q_d X_d \tag{5}$$

When the substitutions indicated in equations (3), (4), and (5) are made in equation (1), and the resulting equation is solved simultaneously with equation (2), we get

$$(Q_c + Q_d)X_o = Q_c X_c + Q_d X_d \tag{6}$$

which is readily simplified, by multiplying out and collecting like Q-terms, to

$$\frac{Q_c}{Q_d} = \frac{X_d - X_o}{X_o - X_c} = \frac{\overline{od}}{\overline{co}} \tag{7}$$

which is the "lever law." Expressed in words, the relative amount of each phase is directly proportional to the opposite arm of the "lever."

INSTRUMENTS FOR RESEARCH

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With the modern need for instrumentation in conducting research has come the problem of providing instruments which are quite expensive to researchers who are either not supported by research money or are supported by a limited amount. Although obtaining instruments of this sort is primarily a problem of the educator in a small college, it has become the problem of people who are associated with institutional research projects or industrial research projects accepted by university or college development funds. With these latter two, there are a great many instances when the researcher working on a directed program finds an interesting sideline of investigation that cannot possibly be investigated with the money that has already been assigned for a specific purpose. Like the teacher in a small college, he is confronted with a problem that is interesting to him personally and on which he would like to work but does not have the money for the necessary instrumentation.

The problem of providing instrumentation for those projects which are not supported by some organized foundation has been attacked in a small way by the establishment of the Vernet Research Instrument Foundation under the direction of Antioch College and associated Board of Directors. The Foundation was established in late 1950 and has operated through the year 1951. The basic idea of the Foundation is to obtain general-purpose instruments that can be placed on loan to workers who will have complete control of the instrument during the time for which they have borrowed it. When the project is completed the instrument reverts to the Foundation where it can be loaned to other workers. The repository for returned instruments is the Chemistry Department at Antioch College.

The equipment loaned by the Foundation may be borrowed and retained under the following conditions:

(1) The equipment shall be under the control of a "responsible investigator." The "responsible investigator" is defined as follows and determined by the Board of Directors after appropriate consultation: "A person whose educational background and/or experience has shown that he can use equipment with a great degree of imagination and ingenuity, yet not destroy its usefulness for general work in the same or allied fields."

The procedure in loaning and returning of equipment is controlled by the following conditions:

(2) The project assignment bears a maximum and minimum time limit determined for each project in which the instrument comes fully under the control of the responsible investigator. There is an added agreement that an extension of time is assured if the project is not completed by the maximum time. The time limits will vary from three months to five years, but must be renewed yearly.

(3) The equipment will be available for demonstration by the responsible investigator to students with the investigator acting as a teacher. However arrangements for demonstrations are entirely under the control of the responsible investigator. The use of the equipment in student projects is not permitted unless full training has been given and the project is in charge of a responsible investigator.

(4) For specific cooperative research projects, the equipment may be placed where it can be most efficiently used, possibly outside Yellow Springs. In such cases, one of the cooperating responsible investigators must be a member of the Antioch College Faculty or associated research projects.

(5) Acknowledgment of use of equipment should be given on any published material that results from the use of the equipment.

(6) The responsible investigator should, by his own initiative, give short written reports to the Board of Directors from time to time on the use of the equipment and be available for discussion of what the project involves.

(7) The responsible investigator should keep a case history on each instrument. This must be kept up to date and inspected before and after each return and reloaning of equipment.

The Foundation realizes that certain endowed projects or industrial concerns might at some time wish to borrow this type of research equipment. Under such conditions, rental charges shall be determined by the Trustees of the Foundation based on ability to pay, and all rental charges go into a separate fund for repair, replacement of parts and insurance to cover equipment.

After the starting of the Foundation and loan of instruments early in 1951 another attribute of such a Foundation became evident. It was found that such instruments could be used for small projects on a cooperative basis with one person in control of the particular instrument.

The project started with a donation of $5000 to Antioch College by the Vernay Laboratories, Inc., Yellow Springs. From this money, a flame photometer and complete high-pressure hydrogenation equipment were obtained. The high-pressure hydrogenation equipment was assigned to the Chemistry Department at Antioch College and the flame photometer to the Fels Research Institute for child development.

The flame photometer was to be used on a project

concerning blood chemistry and the construction of an artificial heart by members of the staff of the Fels Research Institute. Since this work was not directly connected with child development, the Fels Foundation could not furnish money for such equipment. During the year 1951 this instrument was used in furthering the work on the artificial heart and in addition was used on a cooperative basis in three other instances: with local physicians in emergency cases where potassium and calcium analysis was necessary, with the Cincinnati General Hospital Department of Obstetrics which was concerned with toxemia of pregnancy, and with the Children's Hospital, Cincinnati, in cases of adrenal diseases. The instrument was made available twice as a demonstration to student groups from Antioch College during a course in instrumentation.

High-pressure hydrogenation equipment, under the direction of the Chemistry Department, was used for conducting high-pressure carbonations of phenols in connection with a Research Corporation Project on the determination of structure of polycyclic aromatic compounds. The equipment was also used in the hydrogenolysis of acetone pyrroles with two different types of catalysts and in the hydrogenation of a number of other pyrroles to the corresponding pyrrolidines. All of this work was done under a Kettering Foundation fellowship at Antioch College. Besides the above research, this equipment was made available to advanced organic chemistry students in connection with student research problems in organic chemistry.

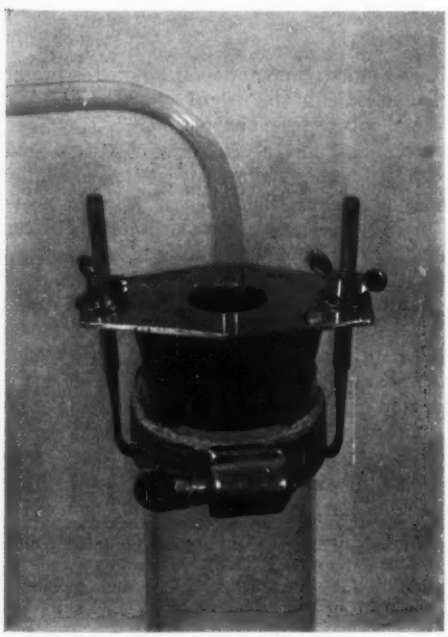
At the beginning of 1952 additional money was added to the Research Foundation Fund and two more instruments have been added which are again general-purpose instruments—a double channel combination magnetic oscillograph and a Lilly manometer oscillator amplifier. These will be used in connection with blood chemistry research at Fels Institute, Yellow Springs.

After the first year of existence for the Research Instrument Foundation, the Board of Directors and Vernay Laboratories have felt that the whole program has been very successful and it is intended that the Foundation Funds be added to every year and additional equipment obtained. Other such Foundations in conjunction with small educational institutions, which make their operations tax-free, would be very helpful in promoting basic research and allowing individual workers to do things that they would otherwise not be allowed to do.

A NEW AND USEFUL LABORATORY CLAMP

ROY L. GRANTOM
Lion Oil Company, El Dorado, Arkansas

QUITE frequently in a chemical laboratory, particularly one in which gas work is being done, the occasion arises when it is necessary to seal a straight-wall glass tube with a rubber stopper so as to allow the maintenance of a moderate pressure in the system.



Since no commercially made clamp is designed for this particular application the usual recourse has been to hold the stopper in place with wire. This method has the obvious disadvantage of uneven pressure distribution and, consequently, a poor rubber-to-glass seal. The clamp shown in the photograph was designed to fill this need.

The clamp consists of a standard rubber hose clamp (such as Arthur H. Thomas Catalog No. 3284), a head-plate, and two connecting bolts with washers and wing nuts. The hose clamp should be large enough to fit around the glass tube and allow some additional space for the insulating tape (rubber or asbestos). The bolts should be approximately $^3/_{16} \times 3$ in. and should have one end flattened and bent so as to fit under the edge of the hose clamp. Those shown in the photograph were made from $^3/_{16} \times 4$ in. toggle bolts. The head-plate can be made of any metal and should be large enough to insure reasonably uniform distribution of the pressure as the plate is screwed down.

The head-plate can be modified as shown to permit the passage of tubes through the stopper.

AURUM POTABILE[1]

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In his book, "Mineralogy and Geology," the great thinker, Johann Wolfgang von Goethe, wrote: "The history of science is science itself." Bearing this statement in mind when reviewing the majority of modern textbooks pertaining to colloid chemistry, medicine and dyeing in regard to what they have to say about the preparation and properties of colloidal gold, it must be admitted that we certainly are still not paying sufficient attention to the importance that the history of science should play in our education.

The major use to which colloidal gold sols, called "drinkable gold" (*aurum potabile*) by the alchemists of the Middle Ages, are put today in the medical field is for the diagnosis of syphilis.[2,3] This test is based on what the colloid chemist calls Zsigmondy's gold number. This is the number of milligrams of protective colloid that, when added to 10 ml. of a standard gold sol, just fail to prevent a change from bright red to violet upon the addition of 1 ml. of a 10 percent solution of sodium chloride. Physicians use this in testing the protective action of cerebrospinal fluid. Unfortunately, this test

FRANCISCI ANTONII
PHILOSOPHI ET MEDICI
PANACEA
AUREA
SIVE
AURO POTABILI,
AD MEDICINALEM POTA-
BILITATEM ABSQUE CORROSIVIS RE-
APOLOGIA
VERITATIS ILLU
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Figure 1

cannot be considered completely reliable; we must bear in mind that it is impossible to standardize gold sols and that the cerebrospinal fluid of healthy individuals can cause the phenomenon of sensitization, which would lead to a faulty diagnosis.

More deplorable, however, is our overlooking the use which *aurum potabile* found as a medication for many diseases two centuries ago. As early as the year 1618 the philosopher and member of the medical profession, Francisci Antonii, published a book[4] about what we today term colloidal gold (Figure 1) in which he discusses in considerable detail the production of colloidal gold sols and their uses for the treatment of various diseases as, for example, venereal diseases, dysentery, epilepsy, tumors, and many others. The author certainly deserves special credit for offering to the reader not only all the favorable medical reports but also

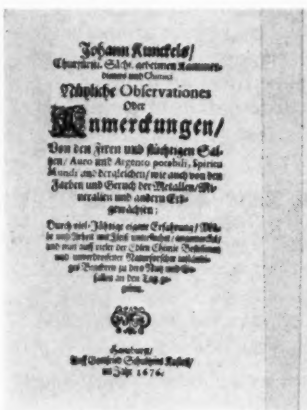

Johann Kunckels
Nützliche Observationes
Oder
Anmerckungen/
Von den Fixen und flüchtigen Sal-
AURUM POTABILE,
oder
Gold-Tinctur,
Dieser
Præparation
Gold Vortrefflichkeit und Analogie
Würckung und Gebrauch
curative & weit als præservative,
Auff viele Curieuse zuthätlich
Hanß Heinrich Heicher,
Breßlau und Leipzig,

Figure 2

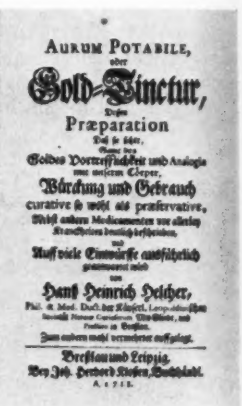


Figure 3

statements made by members of the medical profession who tried to ridicule the use of what they termed "alchemistic, devil-inspired concoctions." He offers quite a few testimonials and case histories of patients suffering from various diseases which he treated successfully with colloidal gold.

In 1676 the German chemist, Johann Kunckels, published a little book[5] on "Valuable Observations or Remarks About the Fixed and Volatile Salts—Auro and Argento Potabile, Spiritu Mundi and the Like" (Figure 2). Chapter 7 is devoted exclusively to "drinkable gold." He points out that the term "drinkable gold" is misleading because it is not possible to dissolve metallic gold except by treating it with acids or melting it with sulfur, and these preparations could not be used as drinkable medications. Since he found, however, that preparations of "drinkable gold" made by others actually contained metallic gold in a neutral, clear,

[1] Presented before the Division of History of Chemistry at the 121st Meeting of the American Chemical Society, Buffalo, New York, March 24–28, 1952.

[2] Glasser, O., "Medical Physics," The Year Book Publishers, Inc., Chicago, **1944**.

[3] Kahn, R. L., "Serum diagnosis of syphilis," J. Alexander's "Colloid Chemistry," The Chemical Catalog Co., New York, **1928**, Vol. II, p. 757.

[4] Antonii, F., "Panacea Aurea-Auro Potabile," Bibliopolio Frobeniano, Hamburg, **1618**.

[5] Kunckels, J., "Nuetzliche Observationes oder Anmerkungen von Auro und Argento potabili," G. Schultzens, Hamburg, **1676**.

slightly pink solution, he concluded that the gold must be present in such a degree of comminution that it is not visible to the human eye. Kunckels certainly deserves far more credit than he has so far received if we bear in mind that his conclusions antedate Michael Faraday's deductions by 180 years and Richard Zsigmondy's visual proof by 230 years. He also stated that these preparations exert curative properties for several diseases.

By far the most complete treatise on colloidal gold was written by Hans Heinrich Helcher, doctor of philosophy and doctor of medicine, at the beginning of the eighteenth century. The title of his book[6] reads (in translation): "Drinkable Gold or Gold Tincture, Its Preparation so that It Surely Describes in Combination with Gold's Excellency and Analogy with Our Body Its Reactivity and Use for Curative and Preservative Purposes, Besides Other Medications for All Kinds of

Figure 4

Diseases Described in Detail and in Reply to Many Objections" (Figure 3). Of the 35 chapters, the titles of the following deserve special attention not only from the point of view of a modern colloid chemist but also by members of the medical profession:

Chapter 1: The preparation of *auri potabilis*.
Chapter 2: The excellence of gold and its value in medicine.
Chapter 5: How this gold tincture in combination with starch should be administered and what diseases it cures.
Chapter 6: How this medicine can be used for preservative purposes.
Chapter 11: That no other metal is comparable to gold and that it helps the patients more than medications obtained from the animal or vegetable kingdoms.
Chapter 12: That the gold tincture actually contains gold and that the starch powder increases its stability.
Chapter 14: That the gold is so small that it can pass through the intestinal tracts of the human body.
Chapter 23: This medicine owes its activity to the gold and not to any added ingredients.
Chapter 31: Although other new medications most frequently have been lost because of jealousy, this one should be retained in unaltered condition for my four sons.

The following words translated from the introduction also deserve special attention:

I would request the impartial reader not to start to mock this book because of its long title and its universally ridiculed name but to suspend voicing his opinion until he has carefully read these pages and has had a chance either to see the effects of this medicine himself or to receive appropriate information from others (Figure 4).

I therefore hope that this treatise will be accepted throughout the world and relieve many suffering from diseases by the use of this medicine and that the pessimists in the medical profession and those who envy me may be converted.

For you, respectful reader, I wish you to receive the same blessing from God as I and other members of the medical profession have now experienced over eight years. If it is God's will then this medicine will be a blessing, disregarding those who envy me, and will act in many cases, even if not with all patients, because disease frequently is God's punishment of those who do not believe in Him or a test for those who are pious.

It would be going too far to offer a complete translation of all the most pertinent parts of Helcher's book, but I would like to state that the author pointed out several times that the use of boiled starch in his "drinkable gold" preparation noticeably enhanced its stability. We therefore cannot deny that he had already noticed the protective action which boiled starch will exert on a colloidal gold sol (Figure 5).

In a French dictionary of chemistry which was published in 1769,[7] the following statement is found under

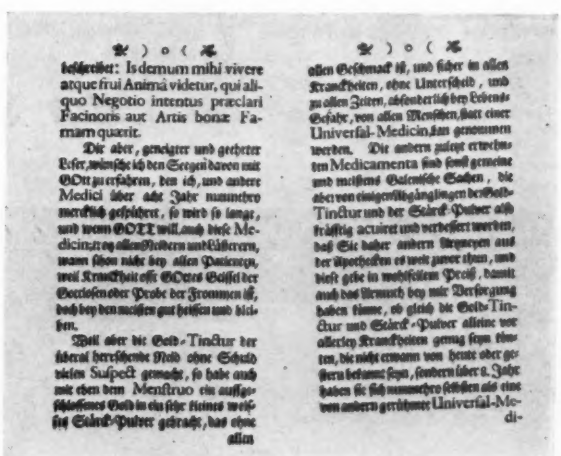


Figure 5

the heading "or potable": "In conclusion it must be stated that medications known as drinkable gold contain gold in its elementary form but of extreme subdivision and suspended in a liquid."

This was the information already available in the eighteenth century. But even over half a century before Thomas Graham coined the term "colloid" in

[6] HELCHER, H. H., "Aurum Potabile oder Gold Tinctur," J. Herbord Klossen, Breslau and Leipzig, 1718.

[7] "Dictionnaire de Chymie," Lacombe, Paris, 1769.

1861 the discoverer of the chemical weights of equivalency, Jeremias Benjamin Richters, had published a book on "New Chemical Facts" in which he devoted a whole chapter to some exceptional properties of gold.[8] Here he offers many suggestions on how drinkable gold can be produced. He even offers an explanation of the differences in color shown by various drinkable gold preparations. He points out that the pink- or purple-colored solution contains gold in the finest degree of subdivision and that the yellow-gold color appears only when the fine particles have aggregated.

From an industrial point of view the early use of colloidal gold as a dye for fabrics also deserves more attention than it has received. In 1794 Mrs. Fulhame published a book,[9] the following quotations from which leave no doubt that she had dyed silk with colloidal gold.

Experiment 1. Gold. I poured a quantity of sulphuric ether on a solution of gold in nitro-muriatic acid, and, by this means, obtained a solution of gold in ether, which I separated from the acid.

In this ethereal solution of gold, I dipped a piece of silk, after it was taken out, and the ether evaporated, it was suspended in a tall glass cylinder, placed over a vessel, containing a mixture of diluted sulphuric acid, and iron filings.

The silk thus exposed to a rapid current of hydrogen gas acquired, after some time, a purple colour, and a large spangle of gold, or an irregular figure, appeared on the upper end of the silk, and looked very brilliant.

The silk was kept exposed to the gas about four months, and frequently observed, but no other remarkable change was perceived, except that the purple colour became more intense; the experiment was now discontinued; and on taking out the silk, and examining it in the light of the sun, particles of reduced gold were observed; but they were very small, and by no means so brilliant, as that, which first appeared.

During the experiment, fresh quantities of material supplying hydrogen gas were occasionally added.

From the brilliant spangle of reduced gold observed in this experiment, I concluded, that there was a sufficient quantity of the metal in the fibres of the silk, could it be reduced.

This spot of reduced gold was very permanent, and adhered firmly to the silk.

Experiment 2. Gold. In order to determine, whether a solution of gold in ether, or one in water, were best adapted to the object of these experiments; I evaporated to dryness a solution of gold in nitro-muriatic acid, and dissolved the salt in distilled water: in this solution, I immersed a piece of silk, which, after it was dried in the air, was suspended in a glass cylinder, like the former piece, and exposed to the action of hydrogen gas about two months.

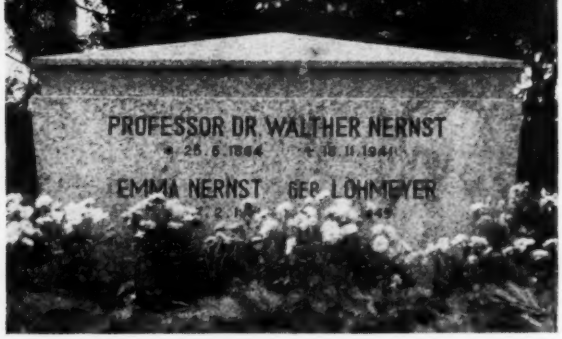
The silk after some time assumed a purple colour, and five or six specks of reduced gold, of the size of pin heads, and one much larger, were observed. Examining the silk in the sunbeams, I perceived the whole of it spangled with minute particles of reduced gold.

After many experiments with these two solutions of gold, I was led to conclude, that the solution in water answered best.

I think that this information will suffice to prove that the scientists of the seventeenth and eighteenth centuries not only knew how to produce what we now term colloidal gold but were also well acquainted with its properties and applicabilities. This information substantiates my opinion that more emphasis must be placed on the history of chemistry in our higher education.

[8] OSTWALD, WI.: "Zur Geschichte des kolloiden Goldes," *Kolloid Z.*, **4**, 5 (1909).

[9] FULHAME, MRS., "An Essay on Combustion with a View to a New Art of Dying and Painting," J. Cooper, London, **1794**.



GRAVESTONE OF WALTER NERNST

WALTER NERNST (b. 1864, Briesen, West Prussia) taught at Göttingen from 1891 until 1905. The remainder of his life was spent in Berlin, where he died in 1941. Early in November, 1951, his ashes and tombstone were brought from Berlin to Göttingen; the urn containing the ashes of his wife was sent from London. Their daughters and a small company of faculty members attended the simple services. The burial plot in the Stadfriedhof is quite close to the grave of Max Planck, which is as it should be since they both contributed so fundamentally to the field of thermodynamics and entropy.—*Ralph E. Oesper, University of Cincinnati, Cincinnati, Ohio.*

BURCKHARDT HELFERICH

RALPH E. OESPER
University of Cincinnati, Cincinnati, Ohio

WÖHLER'S synthesis of urea in 1828 gave the death blow to the "vital force" theory, *i.e.*, it was demonstrated that there is no such fundamental difference between organic and inorganic compounds. Nonetheless, organic chemistry continued to develop separately as the chemistry of substances derived from living nature, a distinction that still holds to a considerable degree. Interest in the processes of the living organism and the substances it produces or elaborates continues to be an important part of organic chemistry. Such problems and the problem of life itself have been the main factors which led Burckhardt Helferich to devote his professional career to chemistry.

Many eminent professors were among his ancestors; his father was a surgeon. (Gottlob Hermann) Burckhardt Helferich was born on June 10, 1887, in the little town of Greifswald, whose university was founded in 1456. After completing the prescribed courses at the local Gymnasium and the "Gelehrtenschule" at Kiel, he entered the University of Lausanne in 1906, where he spent one semester studying science, especially geology. In 1907, after his military service he transferred to Munich and started his chemical training at the University and the Technische Hochschule. He attended the lectures of von Baeyer, and also heard Roentgen in general physics. He was particularly attracted by the vigorous personality of K. A. Hofmann (1870–1940),[1] whom he credits with much of the theoretical and practical foundation of his chemical training. The fame of Emil Fischer,[2] who was then at the height of his career, attracted Helferich to the University of Berlin in 1909. His doctorate was obtained in 1911 under the eminent master, who retained him as his private assistant for a year longer. Dr. Helferich then served as research assistant in the inorganic and organic divisions successively. During World War I he was in the field as an officer. When peace was restored he returned to the University of Berlin, where he habilitated as Privatdozent in 1920. Two years later he accepted a call to the University of Frankfurt as professor of organic chemistry. In 1925 he returned to his native Greifswald as professor of chemistry and director of the chemistry department. He succeeded A. Hantzsch (1857–1935) at Leipzig in 1930. In 1945 Helferich was evacuated to the Western Zone by the American authorities and was welcomed at Bonn as guest professor. When Paul Pfeiffer (1875–1951)[3] retired as *ordinarius* at Bonn, he was succeeded by Helferich. A great part of his time and energy were spent in bringing the Institut back to its proper condition. The building, planned by A. W. Hofmann and first occupied by Kekulé in 1867, had not suffered too much war damage, but the equipment and furnishings were left in very bad condition.

Helferich's independent researches began with his studies of oxyaldehydes, which as intramolecular semiacetals he brought into close relationship with the carbohydrates. He has synthesized several disaccharides and oligosaccharides, *i.e.*, glycosidic condensation products of monosaccharides. Worthy of special mention in this connection are gentiobiose, vicianose, primverose, melibiose, and allolactose. He has made an intensive study of the specificity of emulsin. His method of purifying this long-known ferment is superior to all others; and he has succeeded in dividing this enzyme into several separate ferments. His methods have been extended to the synthesis of other glucosides and to other ferments, particularly the phosphatase of potatoes. He has developed a synthesis of vitamin C and some of its stereoisomers.

The many papers bearing his name, either alone or with those of his co-workers, can be found in the *Berichte*, *Annalen*, *Journal für praktische Chemie*, and Hoppe-Seyler's *Zeitschrift für physiologische Chemie*. He collaborated with Emil Fischer in the 10th and 11th edition of the latter's "Anleitung zur Darstellung organische Präparate" (1922, 1930). He has contributed review articles to the "Fortschritte der Enzymchemie," the "Advances of Carbohydrate Chemistry," and a chapter on "Specificity of the Enzymes" in the recent handbook "The Enzymes."

Professor Helferich's accomplishments have been officially recognized by election to the Akademie der Wissenschaften at Leipzig and the "Leopoldina" at Halle. The Gesellschaft Deutscher Chemiker awarded him its Emil Fischer Medal in 1951.

[1] SCHLEEDE, A., *Ber.*, **74A**, 235–52; SCHWARZ, R., *Z. angew. Chem.*, **53**, 133–5 (1940).

[2] OESPER, R. E., AND DARMSTAEDTER, L., J. CHEM. EDUC., **5**, 36 (1928).

[3] OESPER, R. E., J. CHEM. EDUC., **28**, 62 (1951).

WHAT FACTORS DETERMINE STUDENT ACHIEVEMENT IN FIRST-YEAR COLLEGE CHEMISTRY?

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Is a student's grade in first year college chemistry influenced by whether or not he has had high-school chemistry? By the size of town from which he comes? By his ability as evidenced by his scores on achievement tests? Partial answers to these questions have been obtained by analyzing the records of students in two general-chemistry courses at the University of Wisconsin.

The two courses considered are those for students not planning to specialize in chemistry or engineering. They include students who are preparing for work in various professional fields such as agriculture, home economics, medicine, and pharmacy and also students who are taking chemistry as part of general liberal arts program.

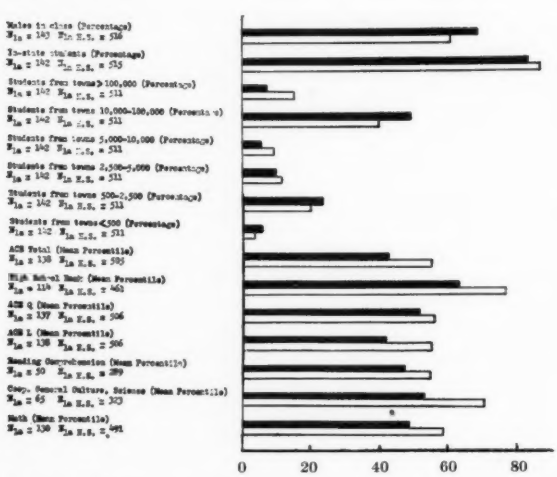

Figure 1. Comparison of Total Chemistry 1*a* Class with Total Chemistry 1*a* H.S. Class

Black bar: Chemistry 1a students
White bar: Chemistry 1a H.S. student

(N_{1a} and $N_{1aH.S.}$ refer, respectively, to the number of students who were enrolled in Chemistry 1*a* and Chemistry 1*a* H.S. and for whom data on the variable in question were available. Because of the lack of complete data, the number of students for whom data for each variable were available is indicated on each graph.)

One of the two courses considered, designated here as Chemistry l*a*, is for students who have had no previous chemistry. It is a five-credit course for one semester in which each student attends two one-hour lectures, two one-hour quiz-discussion sessions, and two two-hour laboratory periods each week. The other course, Chemistry l*a* H.S., is offered for three credits for one semester. In it the students attend two lectures, one quiz-discussion section and one two-hour laboratory period each week. The two courses use the same textbook, cover the same subject matter in lecture, and have identical examinations given at the same time.

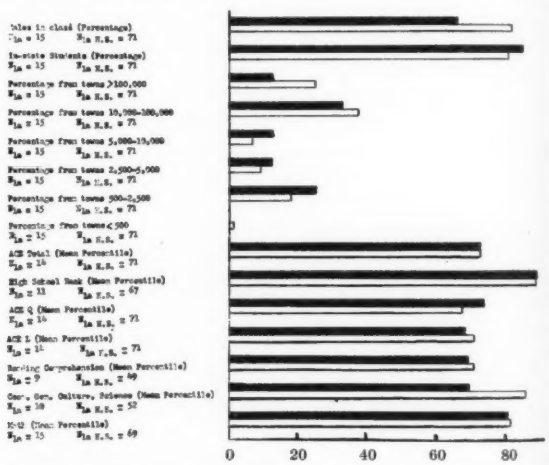

Figure 2. Comparison of Chemistry 1*a* Students Who Received a Final Grade of A with Chemistry 1*a* H.S. Students Who Received a Final Grade of A

Black bar: Chemistry 1a students
White bar: Chemistry 1a H.S. students

The laboratory experiments in Chemistry l*a* H.S. are, in general, chosen to avoid duplication of the experiments done in good high-school courses but to be of the same level of difficulty as those done in Chemistry l*a*. Students from the two courses are not distinguished in the second semester, but enter one course, Chemistry l*b*.

In the organization of the two courses it is sought to allow the student who has had high-school chemistry to prepare himself for the second semester of college chemistry with less investment of time than the student with no chemical background. This saving of time comes primarily in less drill on chemical notation, nomenclature, and the more elementary concepts of chemistry. Other aims of the organizational frame-

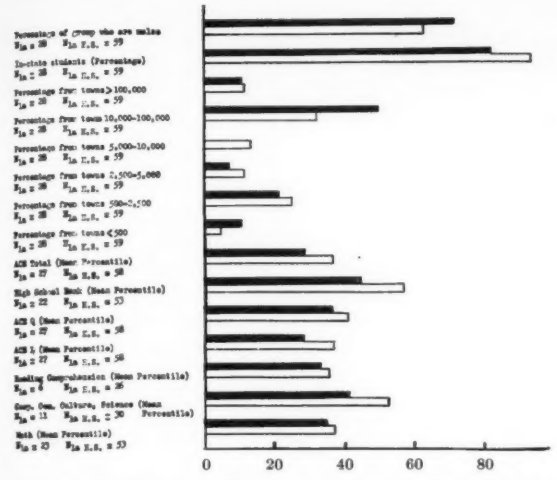

Figure 3. Comparison of Chemistry 1α Students Who Received a Final Grade of F with Chemistry 1α H.S. Students Who Received a Final Grade of F

Black bar: Chemistry 1α students
White bar: Chemistry 1α H.S. students

work are to avoid giving a student full college credit for repeating a subject offered for entrance to the university, to avoid the inferiority feeling with respect to chemistry often felt by students who have had no chemistry when they are placed in a class with those who have had high-school chemistry, and in an equitable way to bring students with and without high-school chemistry to the same point of training at the end of the first semester of college chemistry.

Although the Chemistry 1*a* H.S. students have fewer class hours than the Chemistry 1*a* students, it has been found that they invariably average five to ten points higher on each of the three-hour exams which are given during the semester, and on the final exam. The percentage of final grades of A is higher and of F's is lower in Chemistry 1*a* H.S. than in Chemistry 1*a*, but the average of the two groups together is about the same as in earlier years, when they were both taught in a single 5-credit course.

In view of the markedly higher averages of the Chemistry 1*a* H.S. group, even on the final exam which is given after both groups have completed a semester of college chemistry, we were led to the tentative conclusion that this group must be a select group in some respect in addition to the fact that they have had high-school chemistry. In order to test this hypothesis we have compared the performance of students in both groups, in the fall semester of 1949, with a variety of other data available on the individuals. The broad conclusions are that the students who elect high-school chemistry average higher in general scholastic ability than those who do not, that a student of a given scholastic ability who has had high-school chemistry will make the same grade in Chemistry 1*a* H.S. as a student of the same ability who has not had high-school chemistry will achieve in Chemistry 1*a*, that sex and size of town from which a student comes do not influence his achievement in general chemistry.

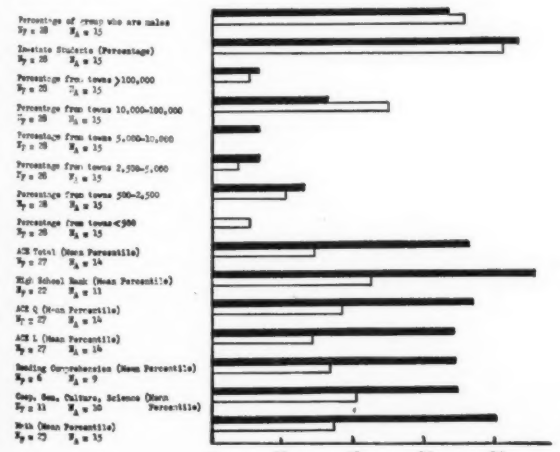

Figure 4. Comparison of Chemistry 1α Students Who Received a Final Grade of A with Those Who Received a Final Grade of F

Black bar: Those who received an A
White bar: Those who received an F

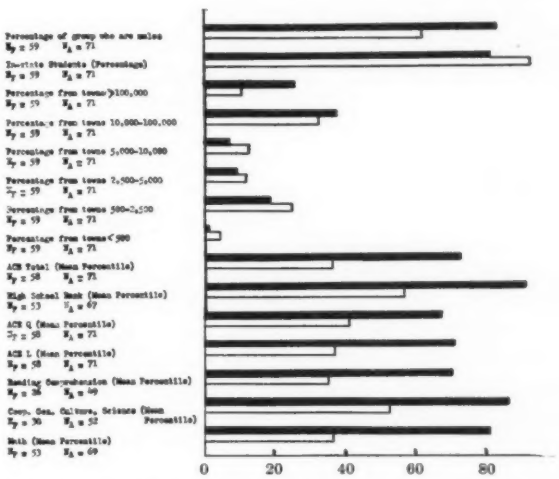

Figure 5. Comparison of Chemistry 1α H.S. Students Who Received a Final Grade of A with Those Who Received a Final Grade of F

Black bar: Those who received an A
White bar: Those who received an F

The two groups, those who took Chemistry 1*a* and who took Chemistry 1*a* H.S., were compared according to the following characteristics: (1) percentage who received each of the final semester grades of A, B, C, D, E, F, and Incomplete; (2) percentage of males and of females; (3) percentage classified as Wisconsin residents; (4) percentages from towns of at least 100,000 population, of at least 10,000 but less than 100,000, of at least 5000 but less than 10,000, of at least 2500 but less than 5000, of at least 500 but less than 2500, and of

less than 500 population; (5) the mean percentile on the total score of the American Council on Education Psychological Examination (ACE)[1,2] (1947 edition); (6) the mean percentile on the linguistic score (L) of the American Council on Education Psychological Examination (1947 edition); (7) the mean percentile on the quantitative score (Q) of the American Council on Education Psychological Examination (1947 edition); (8) the mean rank in high-school class; (9) the mean percentile on the Level of Comprehension score on the Cooperative English Test (Form Q); (10) the mean percentile on the Science section of the Cooperative General Culture Test;[3] (11) the mean percentile on the mathematics section of the Cooperative General Culture Test.[4] The mean (or percentage) of each class was obtained for each of the above characteristics. In addition, mean (or percentage) comparisons were made for each grade category (A, B, C, D, E, F, or Incomplete) for each class.

The table and graphs compare those students who took Chemistry 1*a* with those students who took Chemistry 1*a* H.S. on the above characteristics. Although the data are in some cases too limited to be absolutely conclusive, the tables and graphs strongly suggest the following conclusions:

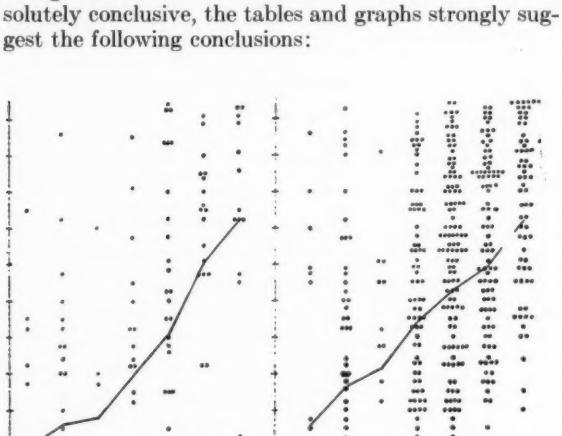

Figure 6. Scatter Diagram Showing ACE Total Percentile Score for Each Student Who Received Specified Final Grades (The line passes through the means for each group.)

[1] The ACE is used at Wisconsin as one indication of a student's scholastic ability. The quantitative (Q) and linguistic (L) scores are combined to get the ACE Total score. The L score, however, is weighted more heavily.

[2] Scores from the American Council on Education Psychological Examination, the Cooperative General Culture, and the Cooperative English Test were obtained from the required entrance examinations for all new freshmen at the University of Wisconsin.

[3] This is one measure of a student's past achievement in science.

[4] This is one measure of a student's past achievement in mathematics.

(1) The students who have taken high-school chemistry and those who have not do not differ significantly in nonintellectual characteristics, such as sex, size of home community, and state residence (Figure 1).

(2) The two groups do differ in intellectual characteristics, *i.e.*, rank in high-school class, the mean percentiles of the ACE total score, ACE Q score, ACE L score, reading comprehension score, score on science achievement, and score on mathematics achievement. The students who have had chemistry in high school are as a group superior in all to the students who have not had high-school chemistry (Figure 1).

(3) There does not appear to be any significant difference between the A students of the two groups except possibly on one score: the science achievement score (Figure 2). The mean score on the science achievement test for those who received A in Chemistry 1*a* H.S. is higher than the mean score for those who received A in Chemistry 1*a*. This difference between the two groups on the science achievement score should be expected since this test was given when both groups entered the University. At the time the Chemistry 1*a* group had not had any formal training in chemistry.

(4) Those students who received F in Chemistry 1*a* and those who received F in Chemistry 1*a* H.S. are below the average of the group on all the intellectual characteristics (Figure 3).

(5) For both the Chemistry 1*a* and Chemistry 1*a* H.S. classes, there is a strong indication that the intellectual variables are highly correlated with the grades received (Figures 4 and 5).

(6) In Figure 2, one will note that 83 per cent of those who received A in Chemistry 1*a* H. S. were males. If the grades were distributed evenly between the two sexes, one would expect that approximately 60 per cent of those who received A would be males and 40 per cent females since that is the sex distribution in the total Chemistry 1*a* H.S. class. In Chemistry 1*a*, 67 per cent of those who received A were males. If the grades were distributed evenly between the two sexes, one would expect that approximately 68 per cent of those who received A would be males and 32 per cent females since that is the sex distribution in the total Chemistry 1*a* class.

The question is then raised why more males, taking into account the sex distribution in each class, received A in Chemistry 1*a* H.S. than in Chemistry 1*a*. Our answer, which is not apparent in the table or graphs, is that the males in Chemistry 1*a* H.S. showed a better performance on some of the intellectual characteristics, which have been shown to be correlated with grades in these chemistry courses, than did the females in Chemistry 1*a* H.S. This difference in performance between males and females was not apparent for the Chemistry 1*a* class. For example, a greater percentage of top scores on the science achievement test, more than would be expected by the sex distribution of the class, was obtained by males than females in Chemistry 1*a* H.S. However, this difference was not nearly as noticeable for the Chemistry 1*a* class.

Comparison of Chemistry 1a with Chemistry 1a H.S. Students

(This table should be read in the following manner: "Total number in class" refers to the total number of students who received the specified grades. "Percentage" refers to the percentage of students receiving the specified grades who belonged to the designated type, *e.g.*, males. For instance, among the Chemistry 1*a* students, of the 17 who received a grade of Incomplete, 76.5% were males. Because of incomplete data the total number of students is not always 143 or 516.)

	Chemistry 1a Students								*Chemistry 1a H.S. Students*							
	Total group	*Inc.*	*F*	*E*	*D*	*C*	*B*	*A*	*Total group*	*Inc.*	*F*	*E*	*D*	*C*	*B*	*A*
Males in class																
Percentage	68.5	76.5	71.0	71.4	75.0	62.9	61.9	66.7	60.7	60.9	62.7	56.2	55.9	56.2	55.4	83.0
Total number in class	143	17	28	7	20	35	21	15	516	23	59	16	93	144	110	71
In-state students																
Percentage	83.8	100.0	82.1	85.7	70.0	85.7	80.9	86.7	86.4	87.0	93.2	93.8	86.0	87.5	83.5	81.7
Total number in class	142	16	28	7	20	35	21	15	515	23	59	16	93	144	109	71
Students from towns > 100,000																
Percentage	7.0	6.2	10.7	00.0	5.0	5.7	4.8	13.3	15.3	14.3	11.9	18.8	9.8	13.3	17.6	25.4
Total number in class	142	16	28	7	20	35	21	15	511	21	59	16	92	144	108	71
Students from towns, 10,000-100,000																
Percentage	48.2	62.5	50.0	71.4	40.0	48.6	47.6	33.3	39.7	38.1	32.2	25.0	44.6	39.9	43.5	38.0
Total number in class	142	16	28	7	20	35	21	15	511	21	59	16	92	144	108	71
Students from towns, 5000–10,000																
Percentage	5.6	6.2	00.0	14.3	00.0	2.9	14.3	13.3	9.6	9.5	13.6	12.5	6.5	9.8	11.1	7.0
Total number in class	142	16	28	7	20	35	21	15	511	21	59	16	92	144	108	71
Students from towns, 2500–5000																
Percentage	9.9	00.0	7.1	14.3	15.0	8.6	14.3	13.3	11.7	14.3	11.9	12.5	15.2	11.2	10.2	9.9
Total number in class	142	16	28	7	20	35	21	15	511	21	59	16	92	144	108	71
Students from towns, 500–2500																
Percentage	23.2	25.0	21.4	00.0	35.0	25.7	14.3	26.7	20.0	19.0	25.4	31.2	20.6	21.0	14.8	18.3
Total number in class	142	16	28	7	20	35	21	15	511	21	59	16	92	144	108	71
Students from towns < 500																
Percentage	5.6	00.0	10.7	00.0	5.0	8.6	4.8	00.0	3.5	4.8	5.1	00.0	3.3	4.9	2.8	1.4
Total number in class	142	16	28	7	20	35	21	15	511	21	59	16	92	144	108	71
ACE Total (Percentile)																
Mean	42.4	23.8	28.8	30.0	39.7	48.1	64.1	73.1	55.5	28.3	36.7	41.4	51.1	57.7	62 6	73.0
Total number in class	138	16	27	7	20	33	21	14	505	23	58	15	91	139	108	71
High-School Rank (Percentile)																
Mean	63.6	49.1	45.9	53.0	59.9	69.4	75.5	91.7	76.4	57.0	56.8	70.5	67.6	78.9	85.9	91.2
Total number in class	114	11	22	7	17	27	19	11	461	21	53	14	82	125	99	67
ACE Q (Percentile)																
Mean	51.1	30.4	36.9	33.4	54.9	54.3	66.1	74.0	55.8	40.4	41.9	42.2	51.1	57.9	61.7	68.0
Total number in class	137	15	27	7	20	33	21	14	506	23	58	15	92	139	108	71
ACE L (Percentile)																
Mean	41.7	23.3	28.1	33.6	34.3	45.4	59.1	68.3	54.9	27.3	37.1	43.5	52.8	57.4	59.9	71.3
Total number in class	138	16	27	7	20	33	21	14	506	23	58	15	92	139	108	71
Reading Comprehension (Percentile)																
Mean	47.3	23.4	33.2	65.7	42.2	42.4	54.7	69.1	54.2	39.3	35.5	25.2	43.3	55.4	60.5	71.2
Total number in class	50	5	6	3	4	16	7	9	289	7	26	8	50	74	75	49
Coop. General Culture, Science (Percentile)																
Mean	52.6	43.4	41.1	41.7	31.4	59.8	60.4	69.6	70.1	59.4	52.1	42.6	59.7	72.5	75.4	86.2
Total number in class	65	5	11	3	8	19	9	10	323	9	30	10	53	87	82	52
Math (Percentile)																
Percentage	48.3	30.5	34.2	31.6	37.4	50.4	66.8	80.4	58.2	41.0	36.3	44.2	46.8	59.2	66.8	82.5
Total number in class	130	13	23	7	19	34	19	15	491	20	53	13	89	137	110	69

(7) In the scatter diagram (Figure 6) one can compare the mean lines of the ACE total percentile score for both classes. There is a direct positive relationship between grades received and ACE total percentile score. Since similar results are obtained when the other intellectual variables are plotted with grades received, only one scatter diagram is presented. Despite the direct relationship, extreme caution must be used when predicting for an *individual* student. For example, note the students in both classes who received an A grade but had rather low ACE total percentile scores.

ACKNOWLEDGMENT

We should like to express our appreciation to Dr. L. E. Drake, Director of the Student Counseling Center of the University, and to the University Computing Service for assistance in this work.

AN INTERPRETATION OF THE PHASE RULE

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THE phase rule can be interpreted as a statement of the *invariance of the number of independent variables* of a system in the following sense: *The number of independent variables necessary to describe a system (with respect to both its extensive and intensive properties) is independent of the number of phases present.*

O. Redlich[1] has taken this interpretation as a basic assumption and has used it to derive the phase rule without use of the second law. However, since the invariance of the number of independent variables is not at all obvious directly, and since it can be derived by use of the second law—that is, by deriving the phase rule in the usual manner—it would seem preferable to think of this invariance as a principal equivalent to the phase rule rather than a basic assumption to be used to deduce the phase rule.

That the principle of the invariance of the number of independent variables cannot be accepted directly can be shown by the following example:

Consider a one-component, one-phase system. The total number of independent variables (extensive and intensive) is three, and these can be taken as the mass, temperature, and pressure. Now suppose that the state of the system is varied so that a second phase appears. How could we know (if we had no phase rule) that this two-phase system would not have four independent variables: the mass of the first phase, the mass of the second phase, the temperature, and the pressure? There is no scientific principle that tells us that this cannot be true—except the phase rule, or some other relationship such as the Clapeyron equation which also depends upon the second law.

As an interpretation of the phase rule the invariance principle is very suitable for presentation to students of elementary physical chemistry. It gives the student a clear picture of the meaning of the phase rule, especially emphasizing the fact that the degree of freedom is a variable which is independent of the masses of the phases. It is easy to visualize and apply a principle that means in effect that for each additional phase present there is one additonal independent extensive variable (the mass of that phase), and, therefore, there is one less independent intensive variable (degree of freedom). From this point of view the phase rule takes on a simple significance and thus becomes more than a mathematical equation to be memorized. The simple examples given later will illustrate this point.

Before considering these examples we will define carefully some important terms and then show that the principle of the invariance of the number of independent variables is indeed a correct interpretation of the phase rule.

DEFINITIONS

Before the phase rule can be discussed from any view point it is necessary to make a clear distinction between intensive and extensive variables, and between chemical species and independent components, and to define precisely the degree of freedom. Although definitions of these quantities are easily found in textbooks and other places (ref. 1), it seems desirable to include them here with some discussion.

Suppose we have two identical systems, and that some property Q of each system has the value q. Now consider the two systems to be one system of mass twice that of each of the original systems.

If the value of Q for the combined system is $2q$, the property Q is an *extensive* property (variable). Examples are volume, entropy, internal energy, etc.

If the value of Q for the combined system is q, the property Q is an *intensive* property (variable). Exam-

[1] REDLICH, OTTO, J. CHEM. EDUC., 22, 265 (1945).

ples are temperature, pressure, concentration, density, etc.

The number of *independent components* of a system in a given state is the smallest number of chemical species that would be required to prepare the system in the given state with masses of the phases in any arbitrary proportions. All independent chemical reactions which are at equilibrium in the system must be considered to reduce the required number of chemical species to the smallest possible value.

That one would not be sufficiently precise by defining the number of independent components simply as the smallest number of chemical species that are required to prepare the system can be illustrated by the following example:

Consider a system composed of one mol of $CaCO_3$ (solid), one mol of CaO (solid), and one mol of CO_2 (gas) all in equilibrium at some temperature, say, 500°C. This is a three-phase, two-component system. However, this special system, with its CaO and CO_2 phases in this particular ratio, could have been prepared from one chemical species—$CaCO_3$. Thus it is necessary to consider systems in which the masses of the phases are in other ratios. Sometimes this difficulty is presumed to be overcome by requiring that *each* phase be prepared *separately*. However, according to such a definition it is not clear that the three-phase system $CaCO_3$(solid) - CaO (solid) - CO_2(gas) is not a three-component system.

The number of independent components may depend upon the state of the system. For example, the one-component, one-phase system $CaCO_3$ is a special case of a system which is usually a two-component system. However, it should be noted that if the solubility of CaO and CO_2 in $CaCO_3$ cannot be neglected, this one-phase system will seldom exist at the exact composition of $CaCO_3$ and will, therefore, be a two-component system.

The *degree of freedom* is a property (variable) of the system which is independent of the masses of the phases.

The degree of freedom is an intensive variable. However, some intensive variables cannot be degrees of freedom according to the definitions given here. For example, the average density (ratio of total weight to total volume) of a heterogeneous system is not a degree of freedom since its value depends upon the relative masses of the phases.

INVARIANCE OF THE NUMBER OF INDEPENDENT VARIABLES

The ordinary phase rule can be written in the form

$$F + P = C + 2$$

where F is the number of degrees of freedom, P is the number of phases, and C is the number of independent components. If the system can exist as one phase we know that the number of independent variables is $C + 2$, namely the mass of each component and two other variables such as the temperature and pressure. We must show that $F + P$ is the total number of independent variables when P phases are present.

The total mass of a phase is an independent (extensive) variable since the mass of a phase can be varied independently, say, by mechanically removing a portion of that phase or adding to that phase from another identical system. The system can have no other independent extensive variables if the temperature and pressure are used as variables since any other extensive variable is determined by the mass of each phase, the temperature, and the pressure. By definition F is the number of independent variables that remain after the masses of the individual phases have been eliminated as variables. Thus, it follows that $F + P$ is the total number of independent variables when P phases are present, and the phase rule states that this is constant (independent of P) and equal to $C + 2$.

EXAMPLES

(*1*) *One-component.* Consider a one-component system, such as pure water. When the system exists as one phase we know that three independent variables are necessary to describe the system with respect to its extensive and intensive properties—for example, the mass, temperature, and pressure. According to the phase rule this number of independent variables is independent of the number of phases.

As long as we consider *all* of the independent variables of this one-phase system, we can use one, two, or three extensive variables as our independent variables. Three extensive variables could be mass, volume, and internal energy. However, if we ignore the mass in order to count the degrees of freedom, the volume and internal energy no longer have meanings, and we must use intensive properties such as molar volume and molar internal energy, *i. e.*, volume and energy per mol. Thus, the degrees of freedom of this one-phase system is always two, regardless of what variables we use to describe the system.

When this system exists as two phases in equilibrium (such as liquid and vapor), there are two independent extensive variables: the mass of the vapor and the mass of the liquid. This leaves only one intensive variable as a degree of freedom.

When the system is vapor, liquid, and solid in equilibrium there are three extensive variables and no degrees of freedom.

(*2*) *Two-component.* Consider the two-component system NaCl and water. The total number of independent variables is four: the mass of the water, the mass of the NaCl, the temperature, and the pressure, for example. The phase rule tells us that this number *four* is independent of the number of phases. Thus, if there are three phases present (say, water vapor, ice, and solution) the mass of each phase can be counted as an independent extensive variable leaving only one degree of freedom as the remaining independent variable. This degree of freedom can be taken as the temperature, or the pressure, or the concentration of the solution, or the density of the vapor, etc.

EARLY METHODS OF SALTPETER PRODUCTION

PAUL C. ZIEMKE
Oak Ridge, Tennessee

HISTORY is repeating itself in the present plan to stock-pile scarce minerals received from foreign nations through the Marshall Plan. World War II taught us an expensive lesson as we found ourselves cut off from strategic items of many kinds, including the precious uranium ores. If the War Emergency Board has its way, we will not be caught napping again.

Our pioneer forefathers also learned the hard way. The War of 1812 and its drastic embargo was forced on them through the bitter struggle between Napoleon and Great Britain. Britain, when hostilities broke out, quickly used her age-old weapon of embargo against us before we drove up prices and depleted the market of scarce materials.

Warfare of that early day was strictly a black-powder operation, an important ingredient of which was saltpeter (sodium nitrate). We had a fair supply of sulfur and an excellent source of charcoal in the form of limitless hardwood forests, yet our saltpeter supply had come from abroad, primarily as ship ballast. The search was on in full force for salt springs or deposits of nitrous earth from which the valuable chemical could be extracted in quantity.

Just who found the saltpeter in the Great Mammoth Cave in Kentucky is not definitely known, but early notes disclose that its presence there zoomed up the price of the land to almost astronomical figures. When war broke out the property changed hands three times in one day. The first sale of 200 acres brought $116; the second, $400; and the final deal of the day, a purchase involving only 156 acres of the orginal tract, was closed for $3000. A bit of work with the pencil will disclose a profit of more than 3315 per cent in one day. A tract of 78 acres sold for $10,000 thirty days later.

Early records indicate that two promoters from Philadelphia, Messrs. Gratz and Wilkins, were reported to have accumulated a fortune through the sale of Mammoth Cave saltpeter. Their resident representative was Mr. Archibald Miller who managed the reduction plant for the elimination of undesired minerals present in the crude deposit.

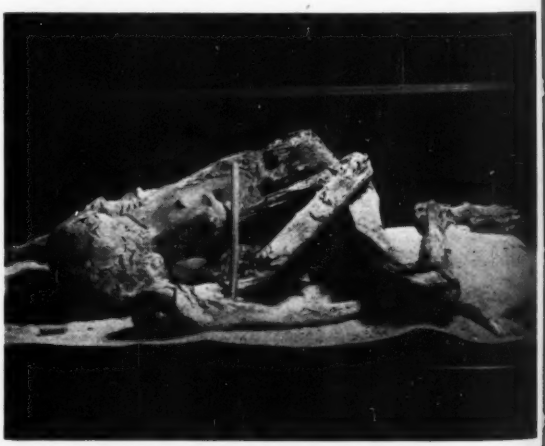
Visitors to the cave may still see the well-preserved installations where with the crudest of implements the miners of that early day worked laboriously, yet effectively, to produce vast quantities of "peter dirt" as it was known in those times. The deposit was not entirely the result of gradual decomposition of the native minerals inherent in the limestone but, like the vast guano beds off the coast of Chile, was built up through countless ages by swarms of bats that numbered well into the millions.

The large openings in the cave made possible the use of oxcarts for the hauling of the peter dirt from the larger rooms to the refining vats, also located in the cave. The smaller caves were exploited by the use of crude, wooden-wheeled barrows that brought the dirt to the main haulage avenues. Cumbersome, inefficient lighting was used in the form of pine knots, whale oil lamps, and tallow candles. Since the caves were chilly to sit in during meal time or rest periods, the miners huddled around wood fires in their sweat-dampened clothes. Wierd indeed must have been the sight of the miners, mostly Negroes, as they drove the oxcarts, wheels squealing, through the dimly lit subterranean passages and tortuous turns.

Just a short distance from the entrance, pits were dug for the leachers, constructed with leaching tanks of pitch-jointed plank. The trunks of cedar or tamarack trees, approximately six inches in diameter, were bored through half-way from opposite ends to form pipes having bell joints, not unlike our present cast-iron water and gas mains.

Considering that these crude pipes measured 20 feet in length, it is most remarkable how straight the early craftsmen were able to guide their long, hand-made augers to have the two bores meet exactly in lines. The thin ends, occasioned by the removal of wood to shape the bell and spigot ends, were reinforced with bands of strap iron, shrunk on while hot.

These effective wood pipes were laid to surface springs to supply water for the leaching tanks farther



Ancient Miner's Skeleton Found Crushed in Cave

Vats Used in Leaching Operation

down in the cave. Main supports consisted of pillars of limestone piled in "dry wall" fashion without the use of mortar. Each pillar was arranged to support two wood lines, one below the other. The upper line pitched down hill to the leaching operations and the lower one was on a grade to return the saline solution to the surface. The feat was accomplished by hand pumping the solution to an elevated tank, establishing the static head required to force it to the evaporators.

Today's visitors to this National Park may still see the well defined ox-cart ruts in the cave floor, since the unchanging temperature has preserved them through the gradual hardening of the spent or lixiviated slimes removed from the leaching tanks. The smoke-smudged walls and ceiling, too, remain to indicate the scene of the feverish activities carried on.

The chemist and engineer will marvel at the ingenuity displayed by the craftsmen in developing the maze of drainage troughs under the vats. They consist of several layers of bored wood pipes, carefully split into halves, or what appears to be natural hollow logs with dry rot scooped out to form troughs by careful splitting. The top layer appears to be a layer of upper-half circles, while the succeeding layer was a row of lower-half circles. The clever feature about this unique construction was the fact that their centers were off the vertical which permitted the lixiviated dirt to settle out of the solution and remain behind.

The saline solution upon arrival at the surface (cave entrance) was handled by a bucket brigade to raise it to an elevated wood tank. From there it flowed to concentrators consisting of open iron vats set up on stone piers above wood fires. The enriched solution was drawn off through a series of troughs filled with wood ashes which added the desired quantity of potassium, an essential ingredient in the making of gun powder. Secondary boiling further concentrated the liquid, after which it was allowed to cool and crystallize.

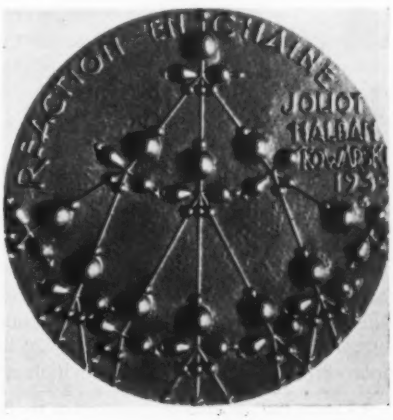
Shipment was made in light cooperage. Horse and ox-drawn wagons hauled it to the warehouse at Green River for eventual transfer to the Ohio River and thence to Pittsburgh and the black powder industry of the Atlantic coast.

With the cessation of hostilities the operations soon fell into decline as foreign saltpeter again was available, especially vast shipments of nitrates from the open pits of Chile. Although more than a century has elapsed since those pioneers labored by the light of pine knots and flickering grease lamps to produce a strategic war material, their installations still remain intact as if they had been left there only yesterday.

As though to guard them from vandalism, not far removed is the mumified form of yet another miner, an aborigine, the "Warrior Mummy" of untold centuries ago, caught in the fall of a huge boulder as he labored to chip out a bit of decorative gypsum from the wall with a crude mussel-shell spoon.

Obverse

A recently designed French medal in bronze (68 mm.) on *Énergie atomique* by the French medalist A. Galtié. It may be purchased from the Administration des Monnaies et Médailles, Paris.—*Herbert S. Klickstein, Edgar Fahs Smith Collection in the History of Chemistry, University of Pennsylvania, Philadelphia, Pennsylvania.*



Reverse

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

INTERESTING PRODUCTS AND NEW DEVELOPMENTS OF THE GENERAL ELECTRIC CHEMICAL DIVISION[1]

KURT C. FRISCH
General Electric Company, Pittsfield, Massachusetts

General Electric has been active in the chemical field since 1900 when the Company established the first industrial research laboratory in the nation. The main efforts at that time were mainly directed at chemical progress as a means of making better electrical products. Taking into account the rapid growth and expansion of chemical products the Chemical Division was formed in 1945 with headquarters at Pittsfield, Massachusetts. Today the Division has four operating departments. They are the Chemical Materials Department with units in Pittsfield, Massachusetts, Schenectady, New York; and Anaheim, California; the Plastics Department in Pittsfield, Springfield, and Taunton, Massachusetts, and Decatur, Illinois; the Laminated and Insulating Products Department in Schenectady and Coshocton, Ohio; and finally the Silicone Products Department in Waterford, New York.

Plastics materials in form of resins or in form of finished products and products closely allied to them, such as surface coatings and plasticizers, represent the major portion of the business done by the Chemical Division, and I will discuss primarily some developments in these fields.

Both thermoplastic and thermosetting materials are being either manufactured or fabricated by G. E. The term thermoplastic indicates, as the name implies, that the material softens under the influence of heat and therefore products made of thermoplastic materials can be remolded.

The principal thermoplastic materials are polystyrene, vinyls, acrylics, cellulosics, polyethylene, and polyamides (Nylon).

Both injection molding and extrusion of thermoplastic materials is carried out by G. E. Injection molding, which is by far the most popular method for molding thermoplastic materials, is the process in which the molding compound is made plastic in a heating chamber from which it is forced by high pressure and introduced into a closed mold which is cooled by means of circulating water. The pressure applied to the compound in the heating cylinder is transmitted to the mold after it has been filled. The pressure is held until the compound has solidified. Then the mold is opened and the pieces are removed.

Examples of injection molded thermoplastic products are television screens and refrigerator inner doors made of polystyrene. The latter are probably the largest existing injection molded parts in the world. Nylon is also manufactured in the form of injection molded pieces, such as the gears illustrated. Nylon possesses some advantages of both the thermoplastic and thermosetting molding materials by being heat resistant (some moldings are dimensionally stable even above 400°F.) and extremely tough at the same time. While the cost of the molding powders is at present relatively high, Nylon can be molded in thin sections which are very strong and, combined with their shorter molding cycle, the cost factor can partly be countered by these properties.

Extrusion is the process of rendering a material plastic and then forcing it through a die and producing tubular or solid articles having the cross sections of the shape of the die opening but indefinite in length. A material frequently used is polyethylene. Like polystyrene it has excellent electrical properties and its chemical inertness and toughness make it a material of a wide range of usefulness. Typical applications include insulation for radar and coaxial cables, food packaging, and many others. At present G. E. is running tests on a farm using polyethylene tubing to pump water from a well and it does not take too much imagination to picture the use of plastic tubing, either of polyethylene or similar materials, for transporting various gases and fluids, in radiant heating, and in many other applications.

In contrast to the thermoplastic materials thermosetting means that heat sets this type of material or, in other terms, when these materials are subjected to heat and pressure, a chemical change "curing" takes place. Once a thermosetting material is cured it cannot be

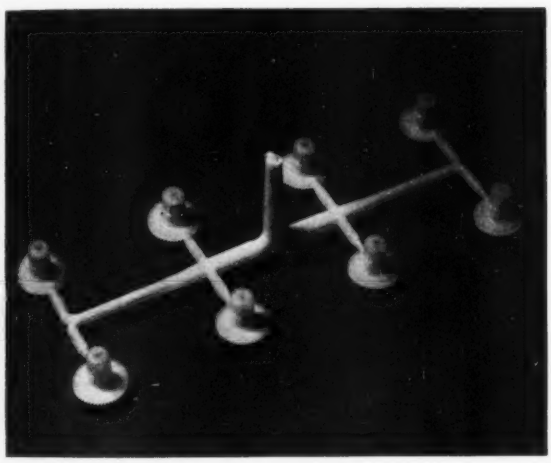
[1] Abstract of an address presented at the 265th meeting of the NEACT, Deerfield Academy, Deerfield, Mass., April 12, 1952.

changed back into its original condition. The principal kinds of thermosetting resins are the phenolics, ureas, melamines, alkyds, certain polyesters, and silicones. Of these G. E. manufactures phenolic, alkyd, polyester, and silicone resins while it only processes the other resins.

Phenol-formaldehyde resins represent a major portion of the thermosetting business. Molded applications such as radio cabinets, toaster bases, camera housings, etc., have grown to include bigger and bigger applications such as television cabinets or the new air-conditioning housing shown in the figure. This is made of woodflour-filled general purpose phenol-formaldehyde compound.

A great step forward was made when Hycar[2], the synthetic rubber made by copolymerization of butadiene and acrylonitrile, was incorporated into phenolic resins. The rubber increases the resilience and shock resistance of the compound considerably and molded applications are particularly well suited for uses requiring exceptionally high impact resistance. Applications of these modified phenolics include soldering guns, various tool handles, fanbases, impellers for automatic dishwashers, and many others.

Window Air-Conditioning Housing Made from Phenolic Molding Compound



Injection Molded Nylon Gears for Oscillating Fan. Cost Reduction Is Effected by Replacement of More Expensive Metal Gears.

So far I have mentioned only molded applications of phenolics. Another important form in which phenolics, or for that matter, other plastics occur, are laminates. Thermosetting resins in solution can be used to impregnate cloth, paper, and various other materials. They are then dried and cut into sheets. In this form they are ready for the laminating press which cures several layers of these sheets into one single sheet of laminate.

In addition to industrial laminates decorative laminates take the limelight of attraction. Urea and melamine resins are often used in conjunction with phenolic resins to produce decorative laminates such as the brand-new development, the "Monotop" laminates. In contrast to older kitchen counter tops or table surfaces, Monotop resists severe abrasions and there is very little in normal everyday kitchen usage that will so much as dim the beauty of the Monotop surface.

Another form in which phenolic resins are being utilized are in surface coatings. It has been estimated that approximately six billion dollars are lost annually in this country because of corrosion and this situation is the more serious in view of the tight metal situation. R-108, a new concept in protective coating intermediates, was developed as the result of a study of the basic processes involved in the phenol-formaldehyde con-

Silicone Oils Flow Freely While Petroleum Oil (at Left) Solidifies Completely at Low Temperatures.

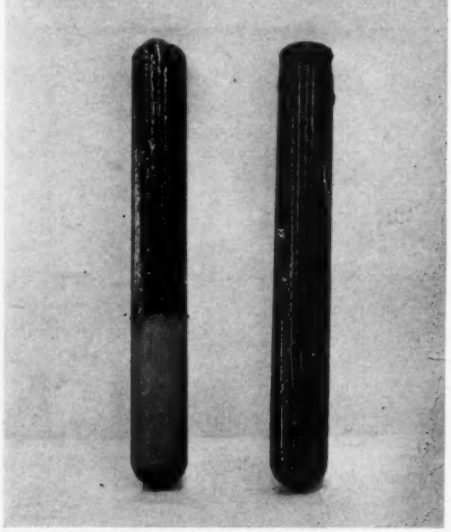
[2] B. F. Goodrich Chemical Co., Reg. U. S. Patent Off.

densations. It combines excellent acid and alkali resistance with toughness, flexibility, adhesion, and heat resistance and may be formulated in a variety of coating applications.

An interesting application of phenolic resins which is growing in ever increasing popularity is as a binder for sandcores which are used in the manufacture of castings. It is based on a new foundry process developed in Germany in the late forties, and is called Shell Molding. Only a relatively small amount of resin is necessary to give the sandcore the desired consistency. The sandshells into which the molten metal is poured can be produced at a faster rate and advantages include savings in sand, metal, and labor besides the fact that closer tolerances can be met. Some forecasts indicate that this application may grow in the next ten years to such proportions that more phenolic resins will be used by it than for plastics molding compounds.

An unusual application of phenolics is G. E. phenolic foam. It is a cellular thermosetting plastic product produced from special phenolic foaming resins for use as a thermal and acoustical insulator. It combines excellent insulating properties with light weight. It is sold commercially in form of liquid resins for "foaming-in-place" operations. Properly activated this resin expands up to 150 times its original volume, solidifying almost immediately to a rigid, cellular structure which fills completely the space in which it is formed. This self-fabricating property enables it to be made in intricate shapes as well as standard block forms.

Among the most interesting products which recently made headlines are certain polyester resins. They are usually made from ethylene glycol and maleic and phthalic anhydrides in various proportions and are then crosslinked with diallylphthalate or styrene. These polyesters are primarily used for the manufacture of so-called "reinforced plastics." In many respects these materials are the glamour products of the plastics industry. They soared into prominence during World War II when glass-reinforced polyesters were used to manufacture helmets and other military applications. Glass mat is put around a mold over which the resin is poured. It is then allowed to cure at room temperature. Afterward the finished board is sprayed to give it the desired color shade. The same type of materials can be used in the manufacture of boats, luggage, and even automobile bodies. At the recent National Plastics Exposition in Convention Hall, Philadelphia, a plastic auto body was displayed, made of glass-fiber reinforced polyester resins. It was built by simply putting a few layers of Fiberglass fabric into a hollow mold, painting on 15 gallons of the resin, and letting the thin shell harden overnight. The body is said to be dent- and rust-proof, and, for its weight, stronger than steel. If there is a break in the body caused by a heavy collision, the repair work can be done easily by the motorist himself by simply applying some glass mat and resin on that spot and smoothing the patch with squeegee. The patch is then painted. The cost of such repair work is only nominal.



Comparison of Two Coated Steel Test Probes After Exposure to 16 Per Cent Caustic Solution at 195°F. The One on the Left Was Covered with a Conventional Phenolic Coating, Lasted but 30 Minutes. The One on the Right, Coated with R-108 Coating Intermediate, Showed No Deterioration at the Time the Test Was Discontinued Five Months Later.

Despite this happy outlook it will probably take some time before the automobile industry, which has invested a considerable amount of money in machine tools, dies, and skilled labor, will change over to plastic bodies.

A relatively new field, at least so far as commercial exploitation goes, are the silicones. Today silicones are prepared mainly by the "direct process," developed by E. Rochow. This process consists of reacting alkyl or aryl halides, principally the chlorides, with a mixture of powdered silicon and metallic copper or silver as a catalyst. When using methyl chloride the predominant reaction can be represented as follows: In addition to

$$2\,CH_3Cl + Si \rightarrow \underset{(I)}{(CH_3)_2SiCl_2}$$

the dimethyldichlorosilane (I) other chlorosilanes such as $(CH_3)_3SiCl$ and CH_3SiCl_3 are formed in this reaction. The chlorosilanes are then hydrolyzed to yield at first silanols (II) and siloxanediols (III) which then can be condensed to polysiloxanes (IV) which are termed commercially silicones:

$$(CH_3)_2SiCl_2 \xrightarrow{H_2O} \underset{(II)}{(CH_3)_2Si(OH)_2} \rightarrow$$

$$(CH_3)_2Si(OH)-O-Si(OH)(CH_3)_2 \rightarrow [(CH_3)_2SiO-]_n$$

(III) (IV)

The outstanding properties of silicones are resistance to high and low temperatures (they resist temperatures as high as 550°F. and as low as − 120°F.,) outstanding release from sticking, unusual surface characteristics,

and inertness. The principal forms in which silicones occur are silicone rubber, water repellents, silicone resins, and silicone oils and greases.

You are probably familiar with the clown of the silicone family, the bouncing putty, at one time merely a laboratory curiosity, but now going into the center of golf balls as well as in automatic leveling devices for table legs.

Silicone rubber, while exhibiting not as great physical strength and abrasion resistance at ordinary temperatures as most natural and synthetic rubber stocks, possesses mechanical properties far superior after exposure at elevated temperatures. These properties have led to the use of silicone rubber in lighting equipment, aircraft engines, electrical apparatus, and chemical processing equipment, just to mention a few applications.

The property of silicones to release from sticking is being utilized to release rubber and plastics from molds and bread from baking pans.

Silicone oils exhibit only a small change in viscosity over a wide temperature range. This property can be demonstrated by comparing a silicone oil with a petroleum oil of like viscosity (at room temperature). On chilling these two oils to a temperature of $-100°$F. the petroleum oil stiffens and congeals while the silicone oil can be poured without any difficulty (see the figure). Silicone oils are being used as damping fluids in internal combustion engines and as lubricating fluids in aircraft gun charging motors among other applications.

I mentioned before the unusual surface characteristics of silicones. One of the most important is water repellency. A blotter partly treated with silicone resin and then dipped in water illustrates this property. A more practical application is the treatment of concrete blocks with silicone resin. Untreated concrete absorbs water easily, but water runs off the treated blocks. This application has of course a big potential market for the protection of residential and industrial buildings.

Resistance to sunlight and weathering in silicones has been utilized in the manufacture of car polishes which are easier to apply and eliminate "rainbow" streaks so common in ordinary car polishes.

Finally, a new development in the mica field will be described. Mica, or more exactly in its form as Muscovite, is an outstanding material in the electrical insulation field. Muscovite, a potassium-aluminum silicate, occurs in two forms, a green and a ruby, the main difference being the higher iron content of the green form. The best grade of mica comes from India while the native mica is usually contaminated with other minerals. G. E. has now brought out a continous sheet of mica, called mica mat, which permits the use of low-grade domestic mica and may free our mica industry from dependence on foreign sources and alleviate a serious shortage threat to the electrical industry. Mica mat is 100 per cent mica in the form of flakes and particles in continuous sheets. It gives better performance than present machine- and hand-laid mica because of its greater uniformity of thickness and void-free construction. Mica mat, when impregnated with resins and bonded to paper, glass, or cloth for greater strength, can be used in form of tapes or sheets in heating devices, molded into shapes for motors and generators, or machine-wrapped on bars and cable.

Mica Mat Emerging from a Machine Where Minute Mica Flakes Are Converted into Continuous Sheets of Mica, Possessing Outstanding Insulation Resistance

In conclusion I would like to point out that the products which I have discussed are merely some outstanding examples of the more recent developments in the Chemical Division. They are the result of a teamwork between research, engineering, and manufacturing which lay the groundwork for the new chemical products of the future.

To the Editor:

Another good and simple temperature-equilibrium demonstration to that given by John A. Brown (J. CHEM. EDUC., **28,** 640 (1951)) is to use a U-tube of nitrogen peroxide. If one limb is warmed the darkening in color is immediately apparent. L. H. ANGUS

BERKHAMSTED SCHOOL
BERKHAMSTED, ENGLAND

To the Editor:

I have encountered this little *problem* which may be of interest to readers.

In order to protect the silverware my wife fashioned some carrying cases out of flannel cloth—a series of pockets sewn together. Each such bundle—of forks, spoons, etc.—could then be rolled up separately. To hold the rolls from unwrapping we put some standard rubber bands around them. Some few days later we unwrapped the silver and discovered that those pieces immediately under the rubber bands bore black striations.

Question: what is the mechanism of this tarnishing?

JULIUS SUMNER MILLER

DILLARD UNIVERSITY
NEW ORLEANS, LOUISIANA

To the Editor:

Miller (J. CHEM. EDUC., **29,** 140–2 (1952)) points out a possible fallacy in the calculation of an over-all redox potential by combination of two half-cell potentials. Discussing the problem of calculation of E°_{1-2} for the over-all reaction

$$2Cu^+ \rightleftharpoons Cu^\circ + Cu^{++}$$

from standard half-cell potentials, he gives, on page 140, three possible solutions (II), (III), and (IV), the first two of which are incorrect, as the author points out, only the third, solution (IV), being correct.

The fallacy of solutions (II) and (III) arise from an incomplete statement and incorrect application of what the author refers to as the "first rule." This "first rule" as stated by Miller is: "A complete chemical reaction is obtained by subtracting one half-reaction from the other; the total e. m. f. for the reaction is obtained by subtracting the corresponding standard electrode potentials."

In order to give a complete statement of this rule, one additional stipulation must be added to the above: the two half-cell reactions combined must correspond, respectively, to the oxidation and to the reduction actually represented by the over-all reaction. In addition, it should be pointed out that it is the standard single electrode oxidation potential of the reduction half-cell reaction that is subtracted from that of the oxidation half-cell reaction, or (the same thing in other words) writing the reduction as a reduction, the sign of the corresponding half-cell potential is changed, this value with sign changed then being added to the oxidation potential of the oxidation half-cell reaction. The latter approach permits the more convenient addition of half-reactions, rather than the subtraction of the half-cell reactions.

In the solution of the above problem, the first step is to divide the over-all reaction into its two component half-cell reactions, one oxidation, one reduction, both corresponding to what actually is indicated by the over-all reaction. Inspection of the over-all reaction shows that one-half of the copper is oxidized from Cu(I) to Cu(II), the other half being reduced from Cu(I) to Cu(O).

Oxidation half-reaction:

$$Cu^+ \rightleftharpoons Cu^{++} + e \qquad E^\circ_1 = -0.168 \text{ v.}$$

Reduction half-reaction:

$$Cu^+ + e \rightleftharpoons Cu^\circ \qquad -E^\circ_2 = -(-0.522)$$

Over-all:

$$2Cu^+ \rightleftharpoons Cu^\circ + Cu^{++}$$
$$E^\circ_{1-2} = E^\circ_1 - E^\circ_2 = -0.168 - (-0.522) = +0.354.$$

In the above, the reduction half-reaction is left as a reduction (the reverse of the oxidation form found in standard tables) so that the over-all reaction is conveniently obtained by addition. To indicate this reversal of direction of reaction, the sign of the corresponding standard single electrode potential is changed.

This solution is, of course, identical with solution (IV) of Miller on page 140. The advantage of the presentation here outlined is that this organization of the solution of the problem shows that this is the only correct solution, and this method automatically precludes the fallacy of Miller's methods (II) and (III). Solutions (II) and (III) are incorrect because each uses the half-cell reaction

$$Cu^\circ \rightleftharpoons Cu^{++} + 2e$$

The fallacy of these methods is that neither the oxidation of Cu(O) to Cu(II), nor the corresponding reduction, is involved in the over-all reaction concerned.

If the single electrode potentials used in either (II) or (III) are the only ones obtainable from tables, then the over-all standard potential, E°_{1-2}, cannot be calculated by subtraction, but these standard single electrode potentials may be combined by the method indicated by Miller in solution (V) in order to obtain the required single electrode potential to be used.

The failure of Miller's solutions (II) and (III) to give the correct answer is not due to any inaccuracy of the rule, but to an incomplete statement of the rule and

a misunderstanding of the application of the rule. Correct application of the rule for combining single electrode potentials to obtain the over-all potential will lead to the correct answer, and preclude the fallacy pointed out by Miller, without resort to the use of volt equivalents. Use of volt equivalents is, of course, necessary in a calculation of a single electrode potential by combination of two single electrode potentials.

J. C. HACKNEY

INDIANA UNIVERSITY
EAST CHICAGO, INDIANA

To the Editor:

The article by Dr. Philip W. West in the May issue titled "The course in analytical chemistry" gives many good values of this course in the chemistry curriculum.

Here in this teachers' training college we find both the qualitative and quantitative courses very helpful to those students preparing as teachers of science in the secondary schools. In these courses the student, preparing to teach the sciences, may learn many important things that cannot be adequately presented in the general chemistry course or by-passed there. This is true since in the general chemistry class we have students preparing to teach home economics, agriculture, and physical education, as well as science majors. In addition to these we have many students taking chemistry to satisfy their science need in "general education."

Since analytical chemistry (which follows our course in general chemistry) is merely an extension of those principles first encountered in general chemistry, it affords a logical place to further elaborate upon those principles or to consider those not previously presented.

It is in the analytical class that the student can discover if he desires to continue as a science major or to go to some other area.

WOODROW W. DORSEY

GRAMBLING COLLEGE
GRAMBLING, LOUISIANA

● ADHESION AND ADHESIVES

Edited by *N. A. deBruyne*, Aero Research Ltd., Duxford, England, and *R. Houwink*, Rubber-Stichting, Delft, Netherlands. The Elsevier Press, Inc., Houston, 1951. xv × 517 pp. 205 figs. 65 tables. 16.5 × 24.5 cm. $10.

THIS book is a substantial contribution to the literature on the subject. Although many books on adhesives have been written, the subject matter treated has been so empirical in nature that it has been difficult to treat it comprehensively and scientifically. In this latest effort by two well-known authors it has been attempted "to deal with the subject in terms of basic scientific concepts. While we appreciated that existing knowledge was too incomplete for such an attempt to be wholly successful, we thought that it might nevertheless still be a significant contribution to knowledge." In line with this laudable objective, the book has been divided into a theoretical part and a technological section. The theoretical aspects were written up first and circulated among the authors of the second section so that they might align their thinking and discussions with it. While the result has fallen short of the editors' hopes, it has made a good beginning in a difficult subject.

The theoretical section has been divided into four main parts, *viz.*, a discussion on the general conditions for wetting and for adhesion by Houwink; a very acceptable survey of molecular forces by A. J. Staverman; that inexhaustible subject, rheology of adhesives, by Hoekstra and Fritzius; and a good contribution on theoretical and experimental studies of stresses in joints by Mylonas and deBruyne.

Part Two, Technological Aspects, has been subdivided into two main sections, organic and inorganic adhesives. The organization of this section has been disturbed a little by treating rubbery adhesives and soldered joints as neither inorganic nor organic. This defect is purely formal, however, and has not reduced the value of the book.

This book could well be used as supplementary reading for courses in colloid chemistry and in fact should be found on the shelves of every university library.

S. S. KISTLER

NORTON COMPANY
WORCESTER, MASSACHUSETTS

● FLUORINE AND ITS COMPOUNDS

R. N. Haszeldine, Assistant in Research in Organic Chemistry, University of Cambridge, and _A. G. Sharpe_, Fellow of Jesus College, Cambridge, and University Demonstrator in Chemistry. Methuen and Co., Ltd., London; John Wiley & Sons, Inc., New York, 1951. 153 pp. 11 × 17 cm. $1.75.

THIS little volume, which is of handy pocket size, aims to present in compact form a fairly complete review of the modern inorganic and organic chemistry of fluorine, with emphasis upon the influence of molecular structure on properties in the inorganic field, and on the effects of fluorine upon the physical and chemical characteristics of organic compounds.

The book is divided into four unequal chapters entitled Fluorine (17 pages), Hydrogen Fluoride (8 pages), Inorganic, and Organic Fluorine Compounds (37 and 84 pages). The first of these deals with the history, preparation, physical and chemical properties of fluorine, together with analytical considerations and a considerable discussion of atomic and ionic structures and sizes, bond distances, melting and boiling points, crystal lattices solubilities and the like, as involved in the various types of inorganic simple and complex salts containing fluorine. The second brief section covers the preparation, physical and chemical properties of hydrogen fluoride, together with its uses as solvent and catalyst for alkylations, acylations, polymerization, and numerous other changes. It also lists many applications of boron trifluoride as a versatile catalyst in organic reactions.

The third portion deals in general with inorganic fluorine com-

pounds classified according to the seven groups and four transition series of the periodic table of the elements. The various simple fluorides are discussed, together with many types of double and complex salts in which the central elements participate in many different valence states. Emphasis is placed upon preparative methods, physical and chemical properties, the progression of these through many series of related substances, electronic structures, crystal lattice configurations, steric considerations, and many other factors designed to present in minimum space the clearest possible picture of the respective compounds. A number of specially interesting features such as acid base reactions in the ionizing solvent bromine trifluoride ($BrF_2^+ \cdot BrF_4^-$) are also presented here. The fourth and final chapter, which constitutes more than half of the book, covers the chemistry of organic fluorine compounds. These are divided into fluorinated haloalkanes, acids, alcohols, ethers, aldehydes, ketones, amines, olefines, aromatic compounds (nuclear and side-chain), and finally the fluorocarbons and other perfluoro compounds. Here, as before, much space is devoted to preparative methods, physical, chemical, and biochemical properties, relative reactivities and tabulations of related data, all well illustrated by numerous examples; together with descriptions of reaction mechanisms (both ionic and atomic), structures, polymeric processes and industrial applications in both the aliphatic and aromatic series, including a considerable discussion of direct and indirect fluorinations, the fluorocarbons and various types of perfluorinated compounds.

For such a compact volume the material is quite well documented by nearly 160 references covering the field up to the summer of 1950, and an adequate subject index is appended.

In the reviewer's opinion the authors have succeeded well in fulfilling their original aim. A large amount of material covering a wide range all the way from theoretical considerations concerning atomic and molecular structures on the one hand, through electronic mechanisms and relative chemical reactivities to preparative methods and industrial uses on the other, have been brought together in a very small space, and little which should have been included seems to have been omitted, considering the size of the book. The organization is good, and the presentation, which is profusely interspersed with illustrative examples throughout, is clear and has been written in such a way as to be understandable to anyone with a sound basic scientific training. Errors in the text are few and entirely trivial in nature, although reference to the "boiling points" of sulfur hexafluoride and cyanogen fluoride, both of which sublime at atmospheric pressure, would seem to be a bit confusing. The binding, printing, and paper of the little book are good.

This volume, obviously intended as a handy working tool, is definitely recommended for the personal library of the fluorine chemist, but in the reviewer's view it should be found more frequently in his coat pocket than on the shelf. Any scientist in need of basic information on fluorine chemistry who looks quickly through its pages is not likely to be disappointed, and students in general should find it a valuable source of information on the subject. Should forthcoming monographs in the series be equally satisfactory, the publishers will have done well in making much scientific material so readily available for everyday use.

LUCIUS A. BIGELOW

DUKE UNIVERSITY
DURHAM, NORTH CAROLINA

● HIGHER CHEMICAL ARITHMETIC

F. W. Goddard, **Senior Science Master, The College, Winchester. Longmans, Green and Co., New York, 1951. viii + 221 pp. 13 × 19.5 cm. $1.60.**

BY THE use of thin (but sufficiently opaque) paper the publishers have compressed the 230 pages of this little book into a thickness of only 1 cm. The author's style is equally economical of space. A very large amount of material is covered with extreme terseness. This book is no substitute for class work in the subjects covered; no space is wasted in explanations for the uninstructed. But it could well be in the pocket of every graduate student studying for comprehensives, for it contains brief, clear, and direct instructions for nearly all conceivable types of calculations involved in (to quote the table of contents) gravimetric and volumetric analysis, solubilities, molecular weights, equivalent and atomic weights, law of mass action, thermochemistry, electrolysis, ionic equilibria, and electrode potentials.

The object of this book is to enable students to pass "the various Higher Certificate and University Scholarship Examinations," and to that end it contains over 400 excellent problems, with answers in an appendix. A strong point in its favor is the complete absence of proportion set-ups; all problems are set up solved for x. A weak point is the use of antiquated methods of writing and balancing chemical equations. Ionic equations are rarely used, and then almost with an apology, while the method of balancing redox equations should be seen to be believed by the younger generation of American chemists. *E. g.*, after giving the following equation for permanganate in acid solution: $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5O$, the author states, "It may help the student to write the key permanganate equation thus: $K_2O.Mn_2O_7 \rightarrow K_2O + 2MnO + 5O$. In modern dress it is, of course: $2MnO_4^- + 6H^+ \rightarrow 2Mn^{++} + 3H_2O + 5O$." The late H. P. Cady was teaching the valence change method of balancing redox equations to freshmen 40 years ago, to this reviewer's personal knowledge. Conceivably our conservative cousins may take note of the ion-electron method some time after the year 2000! But this is incidental; the book does not claim to teach the balancing of equations, and what it does, it does well. The typography is pleasing and no errors were noticed.

EWING C. SCOTT

SYRACUSE UNIVERSITY
SYRACUSE, NEW YORK

● BIOLUMINESCENCE

E. Newton Harvey, **Henry Fairfield Osborn Professor of Biology, Princeton University. Academic Press, Inc., New York, 1952. xvi × 649 pp. 185 figs. 15.5 × 23.5 cm. $13.**

ALTHOUGH bioluminescence has long been a fascinating subject of investigation from various points of view, it is only within the last decade that its outstanding usefulness in the study of fundamental biological problems has been fully appreciated. This usefulness stems from substantial evidence that the intensity of emitted light at any instant is proportional to the reaction velocity of a specific enzyme system. This system, therefore, has a unique, natural indicator of its own reaction velocity, and it has provided an efficient, convenient, and fruitful approach to the kinetic analysis of enzyme activity in relation to numerous factors including, in particular, temperature, hydrostatic pressure, drugs, and different kinds of chemical agents. In other ways also bioluminescence is advantageous in the study of basic problems of cell physiology, biochemistry, biophysics, and other fields. In view of the significance of the work that has been accomplished since the publication of an earlier monograph on the subject by the same author in 1940 ("Living Light," Princeton University Press), this newer and much more comprehensive book is a timely contribution indeed.

"Bioluminescence" is expressly "intended as a complete guide to knowledge on the subject"; a task for which the author is preeminently qualified, and in which he has admirably succeeded. The bibliography of nearly 2000 references gives ready access to all the pertinent literature from about 1800 to 1951 and somewhat later. Moreover, all the important references pertaining to a specific point are conveniently cited together in the text, by author's name and year. Usefulness of the bibliography is enhanced by the inclusion of complete titles and numbers of pages, indicative of the length as well as precise subject. There is no authors' index, but a subject index lists the names of organisms and the chief topics, under a total of some 1500 headings. The voluminous literature that exists on bioluminescence will perhaps be surprising to anyone who has not been actively en-

gaged in its study and has not been aware of its possibilities. In addition to the recent advances, the book makes adequate reference to numerous problems which were touched upon around the turn of the century but which still remain unsolved or call for more critical examination. The accumulated knowledge has been sifted and organized into an impressive compilation of the facts, problems and theories bearing primarily on luminescence, but also on more general problems.

In plan, the various examples of luminescent organisms are considered in phylogenetic order. The 16 chapters are devoted to Bacteria, Fungi, Protozoa and Porifera, Cnidaria, Ctenophora, Miscellaneous Small Groups, Annelida, Mollusca except Cephalopoda, the Cephalopoda, Crustacea, Arachnoidea and Myripoda, Insecta except Coleoptera, the Coleoptera, Echinodermata, Tunicata, and Pisces, in this sequence. Although the higher plants seem conspicuous by their absence, the very recently observed occurrence of detectable chemiluminescence in conjunction with photosynthesis in green plants mean that every major group of plants, as well as animals (short of the higher vertebrates), contain representatives that produce light through metabolic reactions. Among the groups listed above, the taxonomic position of examples known or alleged to exhibit luminescence is indicated, followed by a brief discussion of the various species, their natural history, and the chief facts that have been established concerning the morphology, histology, physiology, biochemistry and biophysics of the luminescent cells, tissues, organs, and in a few intensively studied instances, luminescent extracts. As much information is given as could be feasibly included. While not all of it is equally interesting to specialists in different fields, the enthusiasm, wide experience, and dignified humor of the author are combined to make interesting reading of the factual material as a whole.

The text is generously illustrated with well-chosen figures. A smooth surface paper is used throughout, rendering not only the print and line drawings but also the half-tones pleasingly sharp and clear to the reader. Typographical errors are remarkably few. Considering the immense amount of literature covered, errors of fact are likewise few and minor; *e.g.*, *Sarcina lutea* (page 2) should be *Sarcina noctiluca*, as correctly given in the earlier monograph, "Living Light" (page 6). There are few conclusions or statements pertaining to interpretation which competent authorities might find debatable.

In sum, "Bioluminescence" is a highly informative and scholarly contribution, virtually indispensable to those who are actively engaged with any aspect of the production of cold light by living organisms, and potentially useful to all those whose interests bear on fundamental biological problems, whether from a physiological, biochemical, evolutionary, or other point of view.

FRANK H. JOHNSON

Princeton University
Princeton, New Jersey

● ULTRAVIOLET SPECTRA OF AROMATIC COMPOUNDS

Robert A. Friedel, Physical Chemist, and Milton Orchin Organic Chemist, Bureau of Mines, Bruceton, Pennsylvania. John Wiley & Sons, Inc., New York, 1951. vi + 52 pp. 579 graphs. 20.5 × 27 cm. $10.

Recent years have brought a greatly increased interest in, and use of, absorption spectra for the ultraviolet region of the spectrum. Availability of instruments, such as the Beckman (Model DU) and the Cary spectrophotometers, has facilitated determination of such data. Publication of the results in widely scattered sources, in a disturbing variety of graphical and tabular forms, has made reference work tedious and troublesome.

The general aim of this new compilation was to present, in a consistent plotting form, the data available for 579 aromatic compounds. About half of the curves are from the authors' own laboratory, many previously unpublished, and the others are from the literature. The plotting form used employs log ϵ (molar absorptivity or extinction coefficient) as the ordinate and wave length in angstroms as the abscissa. With the curve are included the name and structural formula of the compound, the solvent used (if known), and the reference (if published). The spectral region covered is mostly the ultraviolet, but it ranges from near 2000 Å. to as high as 5600 Å. for rubicene and 6100 Å. for pentacene. Although contained in a spiral-bound cover, the sheets are punched for a standard 3-ring, loose-leaf book.

The introductory section deals briefly with the following topics: scope of the collection; nomenclature, symbols, and conversion of units; instruments and experimental procedure, incuding spectral band widths used by the authors; theoretical aspects; use of the spectra for qualitative and quantitative analysis; and use of the catalogue. The spectra are indexed both by names and by empirical formulas of the compounds. The latter range from C_5H_5N to $C_{54}H_{38}$.

For workers concerned with the compounds listed, this compilation seems very valuable to the reviewer. He has long wished for such a publication covering colored systems.

M. G. MELLON

Purdue University
Lafayette, Indiana

● THE CHEMISTRY AND ACTION OF INSECTICIDES

Harold H. Shepard, Insecticide Division, Livestock Branch-Production and Marketing Administration, U. S. Dept. of Agricul, ture. McGraw-Hill Book Co., Inc., New York, 1951. vii + 504 pp. 22 figs. 123 tables. 16 × 23.5 cm. $7.

This book is the outgrowth of a mimeographed eidition published in 1939 under the title "The Chemistry and Toxicology of Insecticides." It deals with the chemicals used to control insects rather than with specific insect pests. It discusses insecticides in their chemical, physical, and toxicological aspects, and also presents historical and economic information concerning them.

The author is especially well qualified to write about insecticides. He has experimented with them in the laboratory, lectured about them in the college classroom, and passed judgment upon them as a member of the federal agency which enforces the federal insecticide, fungicide, and rodenticide act. At present the author determines agriculture's need of insecticides, fungicides, and weed killers for the control of various pests, and the figures arrived at are a basis of National Production Authority's action in allocating scarce materials to industry for their manufacture.

The book is divided into 14 chapters which treat of the compounds of arsenic, fluorine, sulfur and copper, miscellaneous substances (compounds of barium, bismuth, boron, mercury, selenium, zinc, etc., and diluents such as clay, talc, and pyrophyllite), plant products such as nicotine, pyrethrins and rotenone, synthetic organic compounds, and attractants and repellents. One chapter deals with the relative toxicity and mode of action of insecticides. Information is given on the effects of insecticides on plants and warm-blooded animals as well as on insects. In the two chapters on synthetic organic insecticides up-to-date information is presented on the newer materials including DDT, benzene hexachloride, chlordan, toxaphene, aldrin, dieldrin, and parathion.

The reviewer heartily recommends this book to all seeking information on the chemicals man must rely upon in his ceaseless warfare against a host of insect enemies.

R. C. ROARK

U. S. Bureau of Entomology & Plant Quarantine
Beltsville, Maryland

● MEDICINAL CHEMISTRY. VOLUME II

Alfred Burger, Associate Professor of Chemistry, University of Virginia. Interscience Publishers, Inc., New York, 1951. xv + 505 pp. (579 to 1084). 1 Fig. 60 tables. 15 × 23 cm. $10.

The second volume of this two-volume treatise includes eleven

major chapters covering the hormones, theories of metabolite antagonism, dyestuffs in chemotherapy, sulfonamides, antimalarials, chemistry of acid-fast infections, metal-free drugs used in tropical diseases, antibiotics, anthelmintics, compounds of arsenic, antimony and bismuth, and antiseptics. Also there are four small chapters dealing with essential amino acids and fatty acids, chemotherapy, antifungal agents, and sterilization and disinfection.

From a chemical point of view this book is well written. Syntheses are clearly presented and structures established. Errors are comparatively few. Nomenclature generally conforms to established rules. Sufficient of the pharmacological data and clinical findings are included to provide chemists with a good appreciation of the medical applications.

As stated above, good nomenclature is generally followed but there are errors. Thus, the one-word name, 1,6-bis-(*p*-hydroxyphenyl)hexane (page 617), contrasts with the two-word name, 3,4-di-(*p*-hydroxyphenyl) hexane (page 612). The latter is incorrect, and unfortunately many such examples occur. Also, one finds ketonitrile (page 597) instead of keto nitrile; or on page 693, arylvinyl ketones instead of aryl vinyl ketones; or page 600, androstenediol-3-acetate-17-benzoate instead of 3,17-androstenediol 3-acetate 17-benzoate. Chemists recognize secondary alcohols but not secondary carbinols (page 615). The author refers to addition of acetylene to a ketone in the presence of potassium amide as the Nef reaction (page 604). To be sure, Nef did carry out this reaction but he was prolific in his discovery of reactions. Other chemists are using the term "Nef reaction" to describe the nitroparaffin-to-aldehyde conversion. At this late date it seems unwise to particularize any of Nef's discoveries as the Nef reaction. One of the few real errors noted in the book is the structure assigned to gamma benzene hexachloride. Instead of all six chlorines being "above" the plane of the ring, two should be below, the two at positions 1, 4. (Bijvoet, *et al.*, *Chem. Abstracts*, **43**, 2838 (1949); **44**, 7614 (1950)). The preferred trivial name of this substance now is lindane, not gammexan.

Antitubercular agents are discussed in the chapter on chemotherapy of acid-fast infections. Naturally, no mention was made of isonicotinic hydrazide since this antitubercular drug was announced after the book was printed, but the omission serves to emphasize the fact that medicinal chemistry is an active and growing field. Burger's treatise is timely. The book can be recommended in high terms as a guide to this territory.

CHARLES D. HURD

NORTHWESTERN UNIVERSITY
EVANSTON, ILLINOIS

● BIOLOGICAL EFFECTS OF EXTERNAL BETA RADIATION

Edited by *Raymond E. Zirkle*, Professor of Radiobiology, Institute of Radiobiology and Biophysics, University of Chicago; Consultant, Division of Biological and Medical Research, Argonne National Laboratory. McGraw-Hill Book Co., Inc., 1951. xxv + 242 pp. Illustrated. 16 × 23.5 cm. $3.25.

THIS volume of the National Nuclear Energy Series includes the usual sixteen pages devoted to forewords, acknowledgments, introductions, and prefaces to which we have become accustomed. It is an account of one portion of the radiobiological program carried out during World War II at the Clinton Laboratories, Oak Ridge, Tennessee. The cover of the book indicates joint editorship by Zirkle and Lawson, but it seems that Lawson was unable to penetrate the binding since he is not mentioned again. The major portion of the investigative work was conducted and reported by John R. Raper.

The first chapter deals with the problems involved in developing an adequate technique for exposing animals to external beta irradiation. The exposure chambers, consisting of pile-activated thick sources of P^{32}, and the ionization chambers used to measure beta-ray intensity and dosage that were ultimately designed are described and illustrated.

The species of animals used in one phase of the work or another were mice, rats, guinea pigs, rabbits, and man. Experiments included the irradiation of restricted areas or the total body surface, and the dose ranged from acutely lethal to minimal levels given in a single or in repeated exposures. A few studies of the effects on the peripheral blood and gross metabolic pattern are presented. The rate of recovery from total-surface beta irradiation and the combined effects of external beta and gamma irradiation also receive consideration. There are excellent descriptions of the acute gross pathological and histopathological changes in the skin.

Those sections of the volume that deal with the delayed effects of single and repeated exposure to external beta rays are not as satisfactory as the reports of the acute lethal work. This situation is probably because the publication deadline antedated the completion of the experiments. Emphasis is rightly placed on the production of neoplasms of the skin. However, the inclusion of a chapter that reviews the literature dealing with the tumor-inducing action of superficial radiations seems to be an attempt to compensate for the incompleteness of the experimental study at the time of writing.

MIRIAM P. FINKEL

ARGONNE NATIONAL LABORATORY
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● QUANTITATIVE ORGANIC MICROANALYSIS

***Al Steyermark*, Microchemical Dept., Hoffmann-LaRoche Inc., Nutley, New Jersey. The Blakiston Co., Philadelphia, 1951. xvi + 389 pp. 155 figs. 12 tables. 15 × 23 cm. $7.00**

BESIDES a description of the expected methods for analysis of the common elements found in organic compounds, Steyermark has included micro methods for phosphorus, arsenic, and oxygen. He has included also chapters on the use of the microbalance, use of manometric apparatus in various determinations, and determinations of neutralization equivalent, alkoxyl groups, acetyl groups, melting point, boiling point, and molecular weight. A chapter on calculations includes the use of factors, finding an empirical formula, tables of gravimetric and volumetric factors, atomic weights and their multiples (to 50 for H), a nitrogen reduction table (logarithms), and a five-place table of logarithms.

Detailed descriptions of the favored methods of analysis are well written from the standpoint of beginning students, who should be able to follow them without difficulty. When more than one method is given, the author frequently makes helpful comments which would assist a reader in making a choice.

The text is very extensively documented with references to original papers: the preface, *e. g.*, having 23 references and the introduction, ten. Sometimes two editions of the same book are listed.

The section in each chapter called "additional information for chapter *n*" is of varied worth. Consider the following four sentences which begin a paragraph, page 74: "Beazley[2] used an electric cumbustion furnace. Hallett[17] used an electrically heated furnace. His apparatus included two jacketed micronitrometers which are used alternately. Royer, Norton, and Foster[43] described automatic combustion." Such disconnected sentences of limited content are scarcely of more value to the reader than a simple listing in a bibliography.

The acknowledgment that microanalysis is in part an art is verified by the vigor with which some practices are condemned by one analyst which are being used and faithfully defended by another. Steyermark's book bears evidence of the art involved throughout the text.

Details are given of many pieces of apparatus which are not available commercially. The diagrams given in the text appear to be cluttered and the heavy typography adds to this impression. Lighter type and better shading would have made the figures much more effective.

There are few errors in printing but a few quirks in English usage occur: "the result would be shy the percentage of 1 carbon atom," page 83; "This must be combusted during the first com-

bustion. . . .," page 107. I consider "combust" a vulgar invention of a verb when we have such a good short one, namely, burn. The author uses "combust" numerous times, *e. g.*, pages 74, 106, 109, 165, etc.

An author and a subject index make the book easy to use.

LEALLYN B. CLAPP

BROWN UNIVERSITY
PROVIDENCE, RHODE ISLAND

THE ENZYMES. VOLUME II, PART 2

Edited by *James B. Sumner*, Laboratory of Enzyme Chemistry, Cornell University, and *Karl Myrback*, Institute for Organic Chemistry and Biochemistry, University of Stockholm. Academic Press, Inc., New York, 1952. xi + 650 (791–1440) pp. Illustrated. 15.5 × 24 cm. $14.

THIS completes the series of four volumes covering many major topics of enzyme chemistry. Consideration of integrated enzyme systems, which had begun in Part I of Volume II, is continued. To the biochemist who is not an enzyme specialist these considerations of mechanisms of metabolic systems involving enzymes may be the most interesting part of the work.

S. R. Elsden discusses bacterial fermentation. F. Bernhein considers enzymes involved in detoxications. H. A. Krebs discusses urea synthesis; P. P. Cohen writes on peptide bond synthesis and on the enzymatic synthesis of glutamine. K.-B. Augustinsson supplies a chapter on synthesis of acetylcholine, cholineacetylase. The enzymatic mechanisms of oxidative assimilation by microorganisms are considered by C. E. Clifton, while S. Ochoa reviews those of carbon dioxide fixation, in a large chapter. F. L. Breusch discusses carbohydrate-fat conversion. W. E. Loomis reviews the present status of theories of photosynthesis in green plants, while C. B. Van Niel discusses photosynthesis by pigmented bacteria.

The Finnish scientists, A. I. Virtanen and N. Rautanen, provide an interesting chapter on nitrogen assimilation by free living microorganisms such as *Azotobacter* and *Clostridium* and in plants by symbiotic microorganisms, also the assimilation of ammonia and of nitrates by plants. The cancer researcher should find interesting a summary of work that has so far progressed in tumor enzymatology. This chapter is supplied by J. P. Greenstein and A. Meister of the National Institutes of Health. There appears to be much more to be learned in this field. Finally there is a long chapter on enzyme technology by the Squibb Institute for Medical Research scientists, A. F. Langlykke, C. V. Smythe, and D. Perlman, ranging from the enzyme technology used in various fermentation of industrial importance, in the production of antibiotics, and in the manufacture and application of all sorts of industrial enzymes such as those important in leather manufacture, brewing, pharmaceutical chemistry, dairy industry, and the fruit-growers industry.

CLIFFORD S. LEONARD

GUILFORD
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ELEMENTS OF CERAMICS

F. H. Norton, Professor of Ceramics, Massachusetts Institute of Technology. Addison-Wesley Press, Inc., Cambridge, Mass., 1952. xiv + 246 pp. Illustrated. 19 × 25 cm. $6.50.

THIS is a book from the pen of an authority. It is intended as an introduction to ceramics for juniors and seniors in undergraduate courses. It should prove useful to workers and others in the ceramic industry.

After a well charted historical introduction which touches upon the scope and magnitude of the industry in the United States and refers briefly to ceramic literature, Chapter 1 on the Clay Minerals opens with the elements of crystal chemistry, followed by silicate structures, formulas and structures of kaolin minerals, montmorillonite minerals, micaceous minerals, and hydrated aluminous minerals, and closes with methods of identification of clay minerals, a diagrammatic summary, and a list of references. Chapter 2 dwells on the Origin and Occurrence of Clays (kaolins, ball clays, fire clays, ferrogeneous and special clays) and considers prices and production. It includes a full-page geologic time scale and numerous maps which show locations of deposits. Chapter 3 is on Properties of Clays. Chapter 4 covers Silica and Feldspar and substitutes for the latter. In Chapter 5 the reader is introduced to Magnesite, Lime, Dolomite, Chromite; tables indicate composition ranges. Chapter 6 on "Other nonclay Minerals briefly considers fluxes like lithium and barium minerals, fluorine and phosphate minerals; the magnesium silicates, talc, and asbestos; aluminous minerals; glaze and glass minerals containing lead, zinc, boron, tin compounds; refractory minerals of zirconium, beryllium, titanium, thorium, cerium, etc.; carbonaceous minerals, graphite, coal and coke, diamond. In Chapter 7, Mining and Treatment of Raw Materials, methods are discussed and well illustrated, including excellent flow sheets. Chapter 8, Flow Properties of Ceramic Pastes and Slips, evidences the author's mastery of applicable physical laws. In Chapter 9 the Plastic Masses appear and in Chapter 10 are used as Casting Slips with process indications. Then follow Chapters 11 on Forming Methods and 12 on Drying of Ceramic Ware. Chapter 13 on Thermochemical Reactions introduces the phase rule, equilibrium diagrams, reaction rates. The reader learns of Types of Ceramic Bodies in Chapter 15, Kilns and Settings in 16. Chapters 17 and 18 cover the Glassy State and Glasses in brief but good fashion, with generous illustrations. Glazes follow in 19 and Enamels on Metal in 20. Chapters 21 and 22 discuss Color Formation in Glasses and Glazes and Ceramic Stains and Colors; then follows Chapter 23 on Decorative Processes. Finally there is Chapter 24 on Cements and Plastics. A useful appendix includes illustrated data on kilns, a section on bodies and glazes with useful charts, brief supplements on colors and stains, tables of equivalent weights, atomic radii, temperature equivalents of Orton cones, a C.–F. temperature-conversion table, calibration data for various thermocouples. The volume closes with a table of symbols and units.

The large clear type is an example of how books should be printed. Italics clearly indicate topics of discussion. Illustrations and tables are abundant and well selected. Many of the drawings were prepared by the author. A glossary of terms might have been helpful to the student. A good book from the pen of an exacting teacher.

ALEXANDER SILVERMAN

THE UNIVERSITY OF PITTSBURGH
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MANOMETRIC METHODS

Malcolm Dixon, Reader in Enzyme Biochemistry in the University of Cambridge, Fellow of King's College, Cambridge. Foreword by the late *Sir F. G. Hopkins*. Third edition. Cambridge University Press, New York, 1951. xvi + 165 pp. 20 figs. 5 tables. 13 × 19 cm. $2.

THE first two editions of this book, during the last 20 years, have served to introduce many workers to manometric methods. The fact that now a third edition has become necessary, shows that it continues to be a favorite among both beginners and experienced. The newest edition is sure to be welcomed by all workers using these methods.

The book, just as in the previous editions, is divided into three parts. In Part 1, the constant pressure, constant volume, and differential types of manometers are described and their theories developed. Some newer improvements have been incorporated. A new Brodie solution and a toluene thermoregulator are recommended, and the newer types of water baths are mentioned. The second part describes in six chapters the methods of measuring respiration. Discussed in detail are the direct and indirect methods of Warburg, the first and second method of Dickens and Simer, and the method of Dixon and Keilin, while the micromethods (*e. g.*, Cartesian diver method, and others) are treated but briefly. A comparison of this part with the corresponding

one of the second edition shows only few changes: newly mentioned are Pardee's method (in a brief footnote only) and also only briefly Laser and Rothschild's new method, both being useful for the measurement of respiration in the presence of carbon dioxide. The third part consists of five appendixes, the first three of which are identical with those given in the second edition (table of physical constants, details of calibration, etc.). Newly introduced, and obviously of great value, is appendix 4, giving nomograms for manometer constants. Appendix 5, equally useful, describes the calibration of interchangeable flasks. The book ends with a list of references, a subject index, and a table of logarithms.

THEODOR VON BRAND

National Institutes of Health
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● CORROSION—CAUSES AND PREVENTION

Frank N. Speller, **Corrosion Consultant. Third edition. McGraw-Hill Book Co., Inc., New York, 1951. xiii + 686 pp. 181 figs. 70 tables. 16 × 24 cm. $10.**

Since 1926, when the first edition appeared, Speller has been the reference book to which chemists and engineers have turned to find a readily understandable explanation of corrosion phenomena and also to find a practical solution to a corrosion problem. The second edition in 1935 is now followed by a third edition in which considerable revision has been made to keep pace with the numerous publications in this field. As with the earlier editions, each chapter has been reviewed by one or more experts in that particular field of corrosion.

The book is divided into two parts; the first covers general principles (296 pages) and the second deals with preventive measures (336 pages). An appendix gives some useful data, test procedures, and glossary of terms used in corrosion. The index is detailed. The first section covers the theories of corrosion; the influence of various internal factors on the corrosion rate, such as methods of manufacture and treatment, and composition of the metal; and the influence of those factors external to the metal. A chapter on principles and methods of corrosion testing concludes the section.

The more extensive section is that on preventive measures, in which is discussed the prevention of corrosion in the atmosphere, underwater, in closed-water systems, in steam generators, in steam and hot-water heating systems, in chemical industries, underground, by stray electric currents, and finally the use of cathodic protection. It is in this section where preventive measures are discussed that Speller is at his best. His long experience as a consultant in this field has given him many examples of unusual corrosion problems and he has had ample time to observe the efficacy of preventive measures.

The book prides itself on its freedom from mathematics and thermodynamics. The "practical" engineer will find a practical solution to many of his corrosion problems, written in a manner he can understand. This book will continue to be the important text and reference book in the field.

KENNETH A. KOBE

University of Texas
Austin, Texas

● ELSEVIER'S ENCYCLOPAEDIA OF ORGANIC CHEMISTRY. VOLUME 14 SUPPLEMENT, PART I, SERIES III, CARBOISOCYCLIC CONDENSED COMPOUNDS. TETRACYCLIC AND HIGHER-CYCLIC COMPOUNDS EXCEPT STEROIDS AND TRITERPENES.

Edited by *F. Radt.* Elsevier Publishing Co., Houston and Amsterdam, 1951. xxxi + 938 pp. (text) + 113 pp. (index). 17.5 × 26 cm. Subscription price, $66. Serial price, $77. Single volume price, $88.

With the appearance of this first supplementary volume to Elsevier's Encyclopaedia, the editor and publishers have honored their initial promise to supply supplements to each volume ten years after publication of the main volume. Volume 14, the first volume of the work to be published, was completed during the war in 1940, although it did not become available until 1946. It covered the literature on higher cyclic condensed ring compounds exhaustively through 1936, with the incorporation of many additional references up to press date. The present Supplement to Volume 14 includes a complete literature survey for the years 1937 through 1946, and, as with the seven volumes previously published, literature concerning the structure of compounds is cited inclusive of the year of publication. There are many literature references for 1950 and 1951 in the present volume.

The choice of carboisocyclic condensed compounds with four rings or more as the subject of the first main volume to be presented has proved to be even more suitable than might have been foreseen, in that a supplement to this volume was urgently needed at this time. At the date of appearance of Volume 14, Beilstein covered cyclic compounds (except for hydrocarbons) only up to 1920. Elsevier only temporarily filled the lacuna in the literature tabulation of these compounds. In the years 1937 to 1946 research in the field was so extensive that even the number of known higher cyclic condensed ring systems increased by sixty per cent (from 233 to 378 systems). Moreover, so much more information became available for the older known systems that, for example, whereas only 14 pages had to be devoted to 1,2-benzanthracene in the main volume, 130 pages were necessary to cover it in the present supplement. The field has assumed such proportions that the supplement to Volume 14 has been divided into four parts. The present volume encompasses neither steroids nor triterpenes. These will be the subject of later volumes to the supplement, two of which are scheduled for publication in 1953.

In compiling the supplement to Volume 14 the editor and his staff have maintained their established high standards of lucidity, critical and intelligent evaluation, careful accuracy, and thoroughness. The literature survey embraces 625 periodicals as compared with 229 periodicals consulted in the preparation of the main volume. Although the patent literature was purposefully excluded from the main volume, it has been covered in the present supplement. In many instances recent unpublished data have been made available through private communications with the investigators. Erroneous and contradictory information in the literature has been corrected, both through the efforts of the Elsevier laboratory staff and with the aid of the authors of the original papers. The very useful summaries of ring systems which precede each structural class of compounds (tetracyclic, pentacyclic, etc.), include all ring systems known up to 1946 with page references to both the main volume and the supplement. In addition, the summary of available methods for formation of the nucleus, presented with most of the ring systems, also refers to both the main and supplementary volumes. This highly helpful cumulative approach to the literature, missing in Beilstein, is, finally, extended to both the subject and formula index in this Elsevier supplement.

Invaluable as Beilstein has been to organic chemistry in the past, the editor and publishers of Elsevier have now shown that a work in the field of scientific documentation superior to Beilstein can be created. Without sacrificing any of Beilstein's recognized thoroughness and rigidly unambiguous system of classification, Elsevier presents a greater abundance of information in a well systematized, more convenient, more stimulating and more concise manner. The emphasis on physical and physiological, as well as chemical data, broadens its usefulness to science in general. The cumulative approach to documentation, the chronological arrangement of literature references after each short section, the mechanical readability, all enhance the attractiveness and usability of this work. For these reasons, and because of its up-to-dateness, it would seem likely that Elsevier will be ranked in first place as a compendium of organic compounds when the main work is complete in 1963.

FRANCES BERLINER

Bryn Mawr College
Bryn Mawr, Pennsylvania

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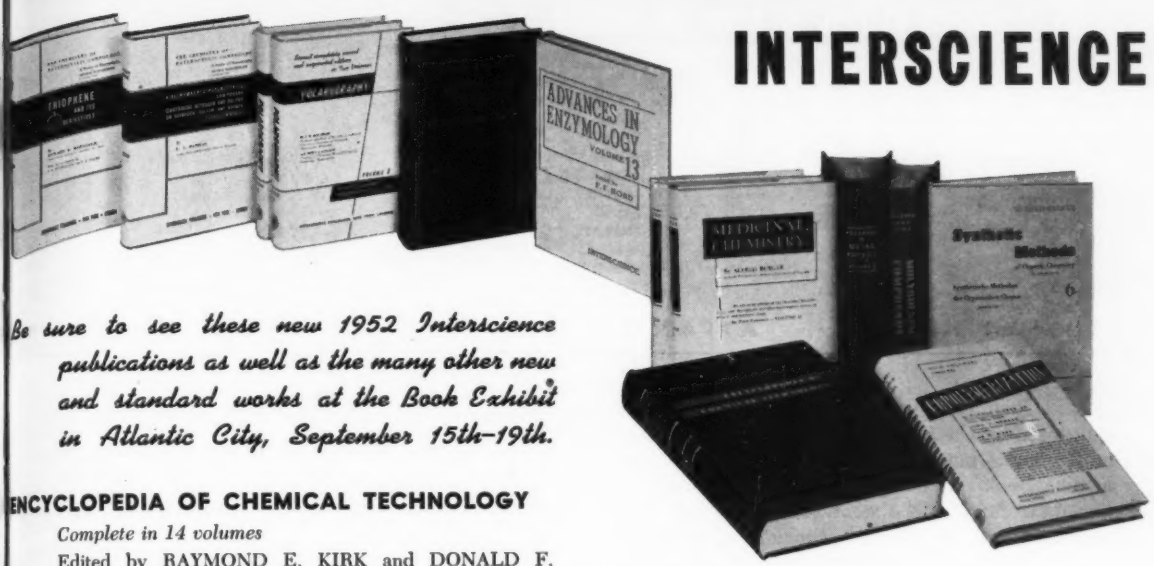

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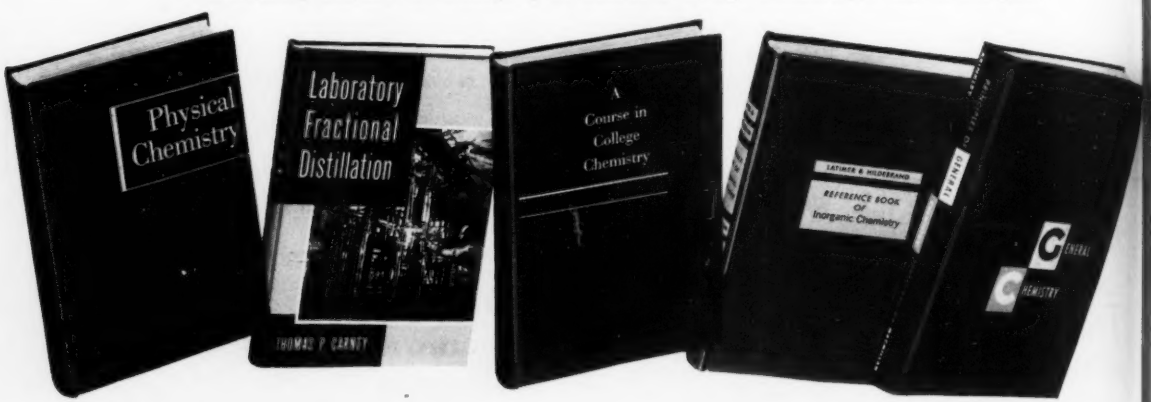

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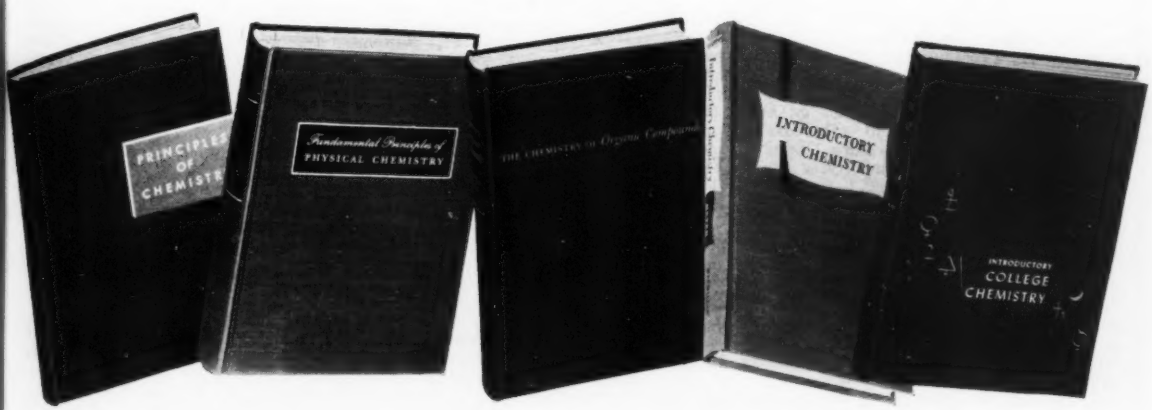

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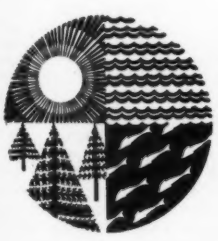

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1952, 300 pages, $7.50

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This volume covers all the fundamental pioneer work of the late W. D. Harkins on the mechanism of liquid/liquid films and interfaces, surface tension and emulsification. Though some of the material has appeared in earlier publications, this new book serves as an invaluable guide to the many advances in emulsion technology—including emulsion polymerization—on which the author was working at the time of his death, much of which has not previously been published. The papers included are offered to the technical public in book form for the first time. All chemists and physicists whose work involves an understanding of detergency, films, foams or any types of heterogeneous systems should find this basic treatise of the utmost value.

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PERFORMANCE OF LUBRICATING OILS—Zuidema

Amply supplied with valuable charts, tables and illustations, this important new book summarizes and evaluates the wealth of data on the performance characteristics of lubricating oils under various conditions. The subject is approached from the point of view of the performance characteristics that are common to all applications. Here, you'll find discussions not only of general lubrication requirements, but *entire chapters* devoted to such important topics as the lubrication process, flow characteristics, sludge and lacquer deposition, emlsification and foaming, with ample reference to viscosity, pour point, and extreme pressure lubrication. The book will prove invaluable to all technical men concerned with the development, manufacture and use of lubricating oils, and to everyone interested in lubrication problems regardless of application.

ACS Monograph, 1952, 250 pages, $5.00

HANDBOOK OF DANGEROUS MATERIALS—Sax

This amazingly complete reference work offers invaluable information to those directly concerned with safety problems: manufacturers, consumers and shippers of chemical products; foremen responsible for safe storage and the overall safety record; and executives who administer the operations. It makes readily available simple, concise and authoritative statements of the hazards involved in thousands of dangerous materials, specific instructions on how to minimize them and useful first-aid information. Also covers explosives, fungus infections and radiation safety. Information includes toxicity, flammability, storage, handling, physical properties and the complete text of the I.C.C. Shipping Regulations which will prove welcome to the many users of this major reference work.

1951, 850 pages, $15.00

PUNCHED CARDS: Their Application to Science and Industry—Casey & Perry

The first complete comprehensive summary of mechanical devices (and punched cards in particular) for scientific information problems is presented in this long-coveted volume. All those concerned with handling large masses of technical data will find the techniques described in this book of utmost value. It describes in detail how to store and codify for immediate reference the vast literature of science through the use of punched cards. The advantages already achieved by using punched cards have resulted in their effective application to an ever-widening range of problems. The purpose of this book is to make existing experience and methods available to all those who deal with scientific and technical information and who must find what they want quickly.

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THE SURFACE CHEMISTRY OF SOLIDS—Gregg

This book is a systematic survey of the various branches of the surface chemistry of solids. It deals with all those chemical and physico-chemical phenomena which relate to the activity of the surface of a solid, or to the interface between a solid and a gas, a liquid or another solid. The author treats many of these phenomena, such as the adsorption of gases and vapors, the spread of a liquid over a solid, catalysis and adhesion, with a consistent viewpoint as far as practicable. He summarizes and evaluates the theories of leading workers in these fields. Emphasis throughout is on general principles in order to simplify an exceedingly complex subject for the benefit of students and others who are not well versed in physical chemistry.

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1950, 630 pages, $12.00

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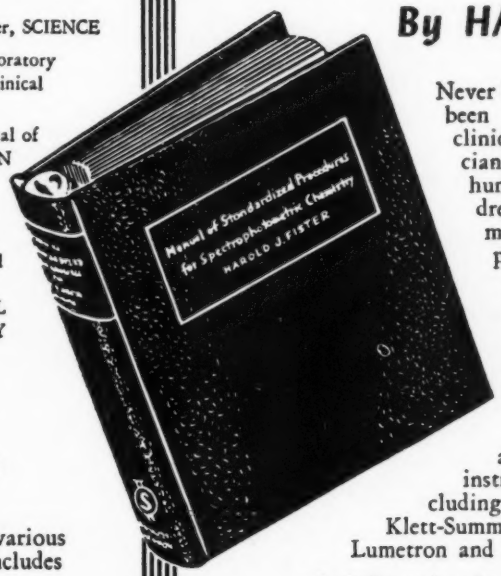

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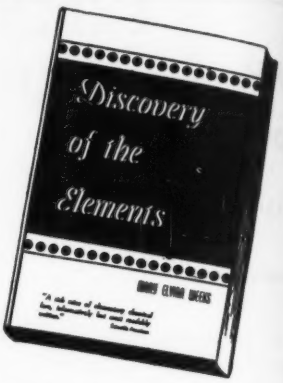

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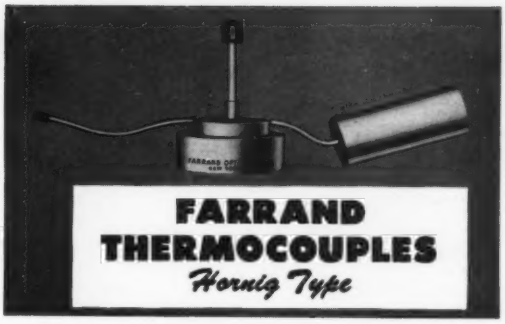

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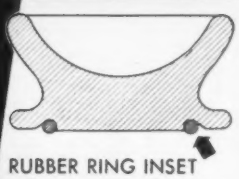

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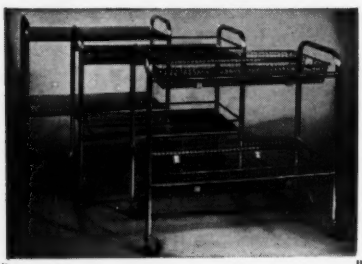

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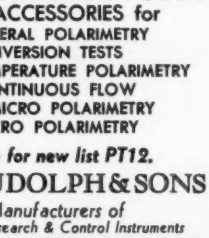

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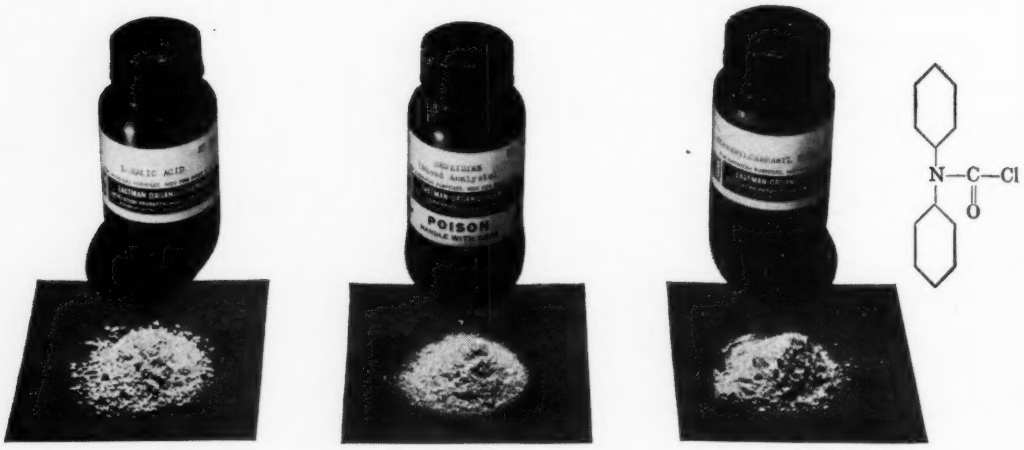

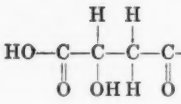

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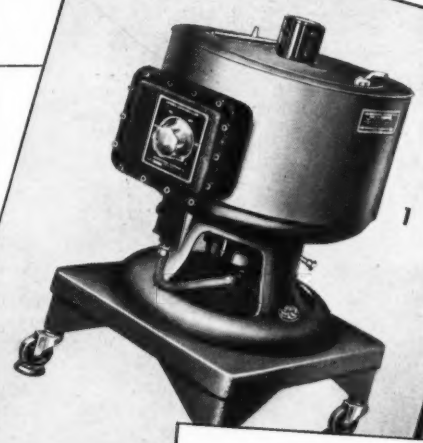

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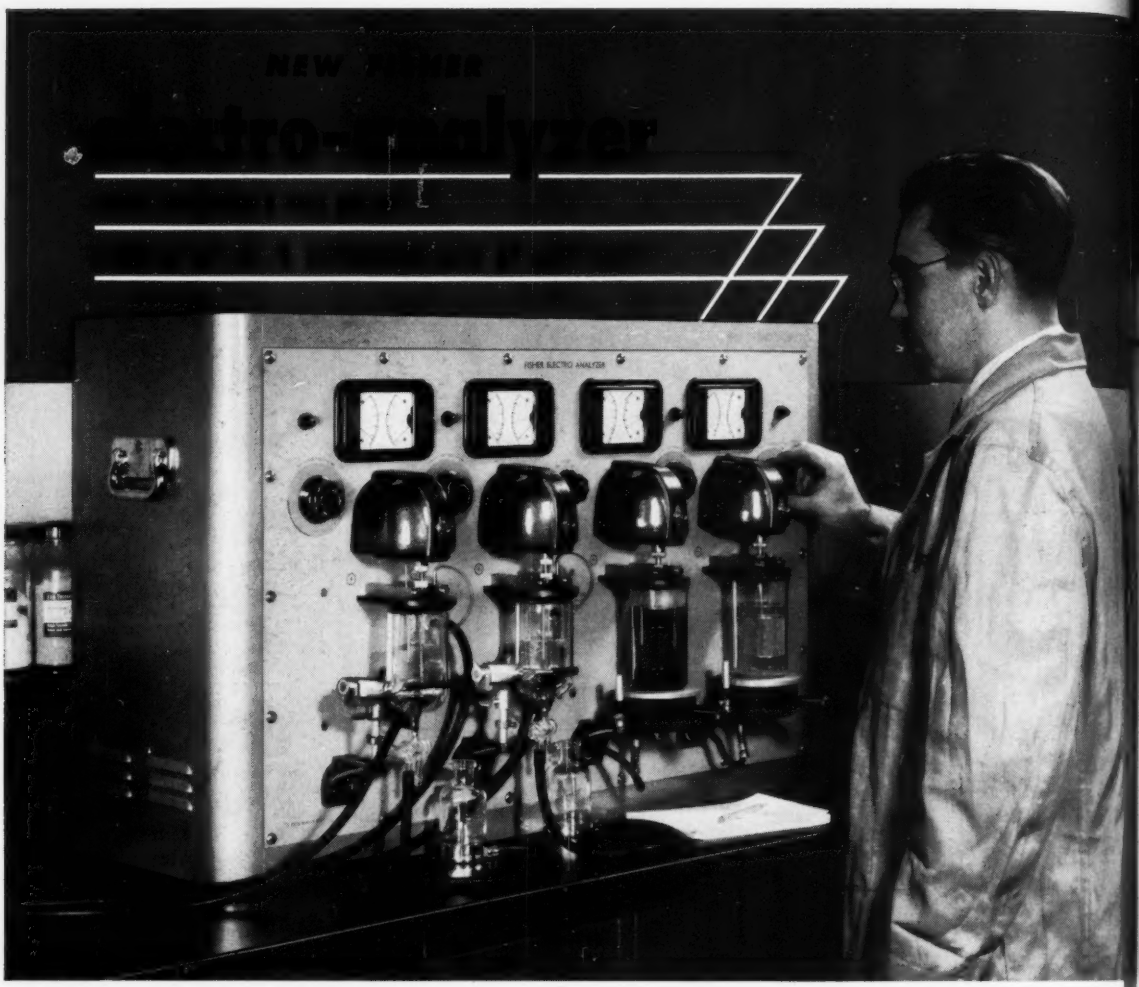

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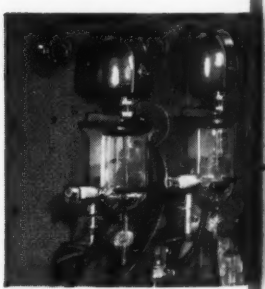

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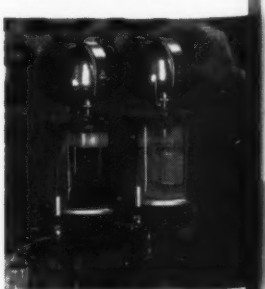

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